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A PRACTICAL TREATISE

FOR THE USE OF

Analytical Chemists, Engineers, Ironmasters, Ironfounders, Students, and Others

COMPRISING

METHODS OF ANALYSIS AND VALUATION OF THE PRINCIPAL MATERIALS USED IN ENGINEERING WORK
WITH NUMEROUS ANALYSES, EXAMPLES
AND SUGGESTIONS

BY

H. JOSHUA PHILLIPS, F.I.C., F.C.S., ASSOC. INST. C.E.

FORMERLY ANALYTICAL AND CONSULTING CHEMIST TO THE GREAT EASTERN RAILWAY
AND SOMETIME CHIEF-ASSISTANT CHEMIST TO THE GREAT WESTERN RAILWAY

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PREFACE.

IN the Author's capacity as an analytical chemist, engaged in the laboratories of railway companies, he has frequently heard it remarked by pupils, whether in chemistry or in engineering, as well as by practising engineers and others, that a work which should give precise methods for analysing and valuing the most important of the materials in general use by engineers, would be of great service. Having himself also felt the want of such a book when a student, he has utilised a portion of his leisure time in collecting the materials for the present work, which he trusts will meet with general approbation, and be found to sufficiently answer the purpose for which it has been written.

It will be observed that Reddrop's "equivalent system" of reagents, of which the Author has now had seven years' experience, is used as far as possible throughout the work. This system he considers preeminently the best for adoption in analytical chemistry, insuring, as it does, accuracy and uniformity of results, and limiting the "personal equation" to a great extent. He is indebted to the courtesy of Mr. W. Crookes, F.R.S., and of Mr. Reddrop himself, for the full description of the system as given by its author in the *Chemical News* of May 23rd and 30th, 1890.

Many of the methods of analysis given in the following pages, as far as the strength and volumes of solutions are concerned, were worked out by the writer when acting as Chief Assistant in the Laboratory of the Great Western Railway, under the direction of Mr. F. W. Harris, F.I.C., to whom he desires to acknowledge his indebtedness for much of his knowledge of chemical analysis. He has also to express his obligations to the authors of the various published works, papers in the Proceedings of Societies, etc., which have been consulted by him, and to which in many cases direct reference is made in the body of the work.

The substance of a smaller work* which the Author had already published, and which has been very cordially received by chemists and engineers, has been embodied, with some slight additions, in this volume (Part III.).

In conclusion, he would say that if the present work prove acceptable, he will gladly avail himself in future editions of any suggestions with which he may be favoured by any of his readers, or for which he may be indebted to notices of the book in the public press.

LONDON,
July, 1891.

---

* "Fuels—Solid, Liquid, and Gaseous: Their Analysis and Valuation."

PREFACE TO THIRD EDITION.

THE Second Edition of this work having run out of print, the opportunity has been taken of bringing the contents more completely up to date. In the result, the work will be found (in comparison with the First Edition of 1891), to have been increased by more than 120 pages, including about 30 new illustrations.

The principal addition to the present issue is the fully illustrated section upon the Microscopical Examination of Iron and Steel, for the substance whereof I am indebted to Mr. J. E. Stead, F.I.C., of Middlesbrough. Other additions include particulars of Hampe's Method for Estimating Oxygen in Copper; Hehner's Bromine Thermal Test for Oils; the Pensky-Marten's Flash-point Apparatus; Redwood's Apparatus for Testing Air for Inflammable Vapour; the Fluidimeter, an instrument of my own design for ascertaining the fluidity or viscosity of several oils at the same time; the Home Office Tests for Determination of Stability of Explosives, etc., etc.

It is hoped that the work, in its present revised and enlarged form, will continue to receive the kindly appreciation of its merits shown by students and others since its first appearance.

LONDON,
December, 1901.

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ENGINEERING CHEMISTRY.

INTRODUCTORY CHAPTER.

REDDROP'S SYSTEM OF REAGENTS.

I THINK it will be generally agreed that many of the text books and standard works on Analytical Chemistry rely rather too much on the discretion of the operator as to the strength and volume of the solutions, &c., which are to be employed in the work of analysis; and if the analyst attempts to follow new methods as he finds them described in scientific periodicals, it is often very difficult to obtain such satisfactory results as the authors of such methods claim for them, owing to the want of a precise and universal system of detailing the *modus operandi* of the processes.

The discordant results of analysis, often obtained by different chemists operating upon the same sample, are much to be regretted; and as the discrepancies are mainly due to differences of working, it is to be hoped that something will soon be done to systematize methods of analysis generally, so that there may be no excuse for differences of result.

A practical insight into chemistry, as affecting engineering, is now considered essential for the complete education of an engineer. As a pupil, the young engineer serves from three to six months in the works' laboratory, and a precise description of the methods of analysis of materials coming under the cognisance of the practising engineer should prove of great service to an engineering pupil, as well as a means of relief to his tutor, who is often preoccupied otherwise than in teaching.

The two main points to be considered in describing a method of analysis are, (1) The *strength* of reagents used; and (2) The *volume* of reagents employed. It therefore becomes necessary to have a stock of reagents of definite strength at hand in the laboratory. As a general rule, in instructions for analysis, the specific gravity of the desired solution or one part of a strong reagent diluted so many times, is referred to as a means of indicating the solution to be employed; but this mode of expressing strength is not very definite or convenient. The best system of reagents with which I am acquainted is that devised by Mr. J. Reddrop, F.I.C., the chemist of the London and North-Western Railway. This system (it will be found) has been followed as far as possible throughout this work, my own experience having led me to fully endorse the claims of its author (see *post*, page 14) as to the advantages to be secured by the employment of the system.

In the year 1877 (Mr. Reddrop states) he introduced into the new laboratory at Crewe a number of reagents, the strength of which was based on the chemical equivalents. Their use was subsequently extended until it included almost all the reagents in the laboratory, and the advantages derived therefrom were found to be so decided, especially in chemical analysis, that they are now employed in all investigations at the Crewe laboratory, and also at the laboratories of the Great Western Railway at Swindon, and the Great Eastern Railway at Stratford.

Subjoined is a full description of the system as given by Mr. Reddrop *:—

Reddrop's System of Chemical Reagents Based on the Equivalents of the Elements.

An equivalent reagent may be defined as one which contains a milligram equivalent of reacting substance in one cc. of solution, or a grm. equivalent in one litre. A solution of this strength is called an equivalent solution, or an equivalent reagent, and is denoted by the symbol E, *e.g.*, E sodium carbonate represents a solution containing 53 m.grms, =0·053 grm. in 1 cc., or 53 grms. in 1 litre. It will be observed that this is not a *percentage solution*, but one containing parts by *weight* (m.grms.) of substance, in parts by *volume* (cc.) of solution. The strengths of all the reagents are expressed in terms of E,* thus the strength of sulphuric acid, sp. gr. 1·84, is approximately 36 E; strong nitric acid, sp. gr. 1·42, is about 16 E; and strong hydrochloric acid, sp. gr. 1·16, is about 10 E. Many of the reagents employed are made of E strength, but in some cases the substance is not sufficiently soluble to make an E solution, and consequently some fractional part thereof has to be adopted instead. Thus we have $\frac{E}{4}$ hydrogen sulphide, $\frac{E}{20}$ calcium oxide, $\frac{E}{30}$ calcium sulphate, $\frac{E}{600}$ strontium sulphate (test for barium). The fractional indices may, if preferred, be expressed decimally, thus $\frac{3E}{5}$ or 0·6 E. All E reagents are equal in precipitating or *quantitative* reacting power. For example, if we have a quantity of lead salt in solution which requires 36 cc. of E sulphuric acid to exactly precipitate the lead salt as sulphate (not allowing for free sulphuric acid in excess), it will require the same number of cc. of E ammonium carbonate to precipitate the lead as carbonate, or of E potassium chromate to precipitate the lead as chromate. The same quantity of lead in solution could, however, be precipitated by 1 cc. of 36 E sulphuric acid, or by 7·2 cc. of 5 E ammonium carbonate, but it would require at least 144 cc. of $\frac{E}{4}$ hydrogen sulphide (saturated solution) to precipitate the lead as sulphide.

---

* Vide *Chemical News*, May, 1890.

It may here be pointed out that $\underline{E}$ reagents are not made with the same degree of accuracy as the *Standard* E reagents employed in volumetric analysis. Variations in strength to the extent of from $\frac{1}{100}$th to $\frac{1}{20}$th part are not considered inadmissible; they may, consequently, be made in almost the same time as those hitherto employed, and the strength, in terms of E, is printed on the label of each bottle, thus—

---

* E being used as the symbol of equivalent or equivalents.

SULPHURIC ACID.
36 E.

or

SULPHURIC ACID.
$36\ \frac{H_2SO_4}{2}$.

or

SULPHURIC ACID.	
36 E.	$E = \frac{H_2SO_4}{2}$.

The accompanying Table includes the reagents to which the equivalent system has already been extended.

Column 1 gives the name of the reagent.

Column 2 the molecular formula of the anhydrous substance.

Column 3 the molecular weight of the anhydrous substance.

Column 4 the equivalent weight of the anhydrous substance.

Column 5 gives the strength of reagent as recommended by Fresenius, "Qual. Analysis," 10th English Edition, first in the terms stated by him, and second, as calculated on the equivalent system. This double column is simply given for comparison.

Column 6 gives the strength of reagent recommended on the equivalent system, first in grms. per litre, and second in terms of E.

Column 7 gives the chemical formula of the substances used, and the methods by which the reagents are prepared of *sufficient accuracy for general use.*

Column 8 gives the strength of reagent as determined by trial after being prepared by the method given.

Several notes are also appended, to which the reader should refer for further information.

NOTES TO TABLE I.

(1) Sulphuric acid, sp. gr. 1·842, containing 98·6 per cent. *by calculation* gives 37·07 E.

Sulphuric acid, sp. gr. 1·84, as supplied, two samples titrated gave 36·6 E and 36·8 E.

(2) I usually make 5 litres of 5 E acid at a time and adjust the strength by titrating 2 cc. with standard N sodium hydrate. This can be done more quickly than by using the hydrometer, owing to changes of temperature on dilution. 5 E ammonium hydrate may be made in a similar manner. These 5 E reagents may also be made by simply diluting the concentrated ones to the right degree.

(3) Nitric acid, sp. gr. 1·50, containing nitrous acid, titrated, gave 22·8 E.

(4) Nitric acid, sp. gr. 1·424, containing 70·2 per cent., by calculation gives 15·87 E.

Nitric acid, sp. gr. 1·42, as supplied, two samples titrated gave 16·05 E and 16·6 E.

(5) Hydrochloric acid. sp. gr. 1·160, containing 31·73 per cent. by calculation, gives 10 08 E.

Hydrochloric acid, sp. gr. 1·16, as supplied, two samples titrated gave 10·36 E and 10·32 E.

(6) Carbonic acid. Water at 15° C. dissolves 1·0020 vols. of CO_2; this, by calculation, gives $\frac{100\ E}{1114}$.

Column 1.	2.	3.	4.	5.	
				Fresenius's System.	
				Strength of Reagent.	
Name of Reagent.	Symbol.	Molecular weight.	Equivalent weight.	As stated.	Calculated in equivalents.
Sulphuric Acid	H_2SO_4	98	49	Sp. gr. 1·840	36 E
,,	,,	,,	,,	1 to 5	6 E
,,	,,	,,	,,	—	—
Nitric Acid	HNO_3	63	63	—	—
,,	,,	,,	,,	—	—
,,	,,	,,	,,	Sp. gr. 1·2	6·16 E
,,	,,	,,	,,	—	—
Hydrochloric acid	HCl	36·5	36·5	—	—
,,	,,	,,	,,	Sp. gr. 1·12	7·48 E
,,	,,	,,	,,	—	—
Sulphurous acid	H_2SO_3	82	41	—	—
Carbonic acid	H_2CO_3	62	31	—	—
Acetic acid	$HC_2H_3O_2$	60	60	—	—
,,	,,	,,	,,	33% of $HC_2H_3O_2$	5·61 E
,,	,,	,,	,,	—	—
Tartaric acid	$H_2C_4H_4O_6$	150	75	—	—
,,	,,	,,	,,	—	—
Citric acid	$H_3C_6H_5O_7$	192	64	—	—
,,	,,	,,	,,	—	—
Oxalic acid	$H_2C_2O_4$	90	45	—	—
Hydrofluoric acid	HF	20	20	—	—
Hydrofluosilicic acid	H_2SiF_6	144	72	—	—
,,	,,	,,	,,	—	—
Hydrogen sulphide	H_2S	34	17	Saturated solution	$\frac{E}{4}$ —
Chlorine water	Cl_2	71	35·5	Saturated solution	$\frac{E}{5}$ —
Bromine	Br_2	160	80	—	—
Bromine water	,,	,,	,,	—	—
Hydrogen peroxide	H_2O_2	34	17	—	—
,,	,,	,,	,,	—	—
Potassium hydrate	KHO	56	56	—	—
,,	,,	,,	,,	—	—
Sodium hydrate	NaHO	40	40	Sp. gr. 1·13 = 9% Na_2O	3·28 E
,,	,,	,,	,,	—	—
Ammonium hydrate	AmHo	35	35	—	—
,,	,,	,,	,,	Sp. gr. 0·96 = 10% NH_3	5·54 E

Column 6. Equivalent System.		7.		8.
Grms. per litre.	Strength.	Symbol of substance taken.	Method of Preparing Reagent.	Strength of Reagent as found by trial.
—	36 E	—	Sulphuric acid, sp. gr. 1·8427 at 15·5° C. Note (1).	36 E
—	5 E	—	Sulphuric acid diluted to sp. gr. 1·1527 at 15·5°. Note (2).	5 E
49	E	—	200 cc. of 5 E sulphuric acid diluted to 1 litre.	E
—	24 E —	—	Nitric acid sp. gr. 1·50. Note (3).	22·8 E
—	16 E	—	Nitric acid sp. gr. 1·4268 at 15·5° C. Note (4).	16 E
—	5 E	—	Nitric acid diluted to sp. gr. 1·1656 at 15·5° C. Note (2)	5 E
63	E	—	200 cc. of 5 E nitric acid diluted to 1 litre.	E
—	10 E	—	Hydrochloric acid sp. gr. 1·1611 at 15·5° C. Note (5).	10 E
—	5 E	—	Hydrochloric acid diluted to sp. gr. 1·0843 at 15·5° C. Note (2).	5 E
36·5	E	—	200 cc. of 5 E hydrochloric acid diluted to 1 litre.	E
—	4 E —	—	Water at 15·5° C. saturated with sulphur dioxide (sp. gr. 1·052).	3·7 E
—	$\frac{E}{10}$ —	—	Water at 15·5° C. saturated with carbon dioxide. Note (6).	—
—	17 E	—	Acetic acid solid at 10° C. Note (7).	16·9 E
—	5 E	—	294 cc. of 17 E acetic acid diluted to 1 litre. Note (2).	5 E
60	E	—	200 cc. of 5 E acetic acid diluted to 1 litre.	E
—	5 E	$H_2C_4H_4O_6$	375 grms. dissolved and diluted to 1 litre.	5 E
75	E	„	75 „ „ „	E
—	5 E	$H_3C_6H_5O_7,Aq$	350 „ „ „	5 E
64	E	„	70 „ „ „	E
—	$\frac{3E}{2}$	$H_2C_2O_4,2Aq$	94·5 „ „ „ Note (8).	$\frac{3E}{2}$
—	12 E ?	—	Hydrofluoric acid, sp. gr. 1·15.	12·9 E
—	—			
—	E			
—	$\frac{E}{4}$ —	—	Water at 15·5° C. saturated with hydrogen sulphide.	$\frac{24E}{100}$
—	$\frac{E}{5}$ —	—	Water at 15·5° C. saturated with chlorine.	$\frac{19E}{100}$
—	37 E	—	Pure liquid bromine. Note (9).	—
—	$\frac{E}{2}$	—	Water at 15·5° C. saturated with bromine.	$\frac{E}{2}$
—	4 E —	—	Hydrogen peroxide, 20 volume solution. [Note (10).	—
—	2 E —	—	„ „ 10 „ Note (11).	—
—	5 E	KHO	280 grms. dissolved and diluted to 1 litre. [Note (12).	—
56	E	„	56 „ „ „ Note (12).	—
—	5 E	NaHO	200 „ „ „ Note (13).	—
40	E	„	40 „ „ „ Note (13).	—
—	20 E —	—	Ammonium hydrate sp. gr. 0·880. Note (14).	18·9 E
—	5 E	—	Ammonium hydrate diluted to sp. gr. 0·9643 at 15·5° C. Note (2).	5 E

Column 1.	2.	3.	4.	5.	
				Fresenius's System.	
				Strength of Reagent.	
Name of Reagent.	Symbol.	Molecular weight.	Equivalent weight.	As stated.	Calculated in equivalents.
Ammonium hydrate	AmHo	35	35	—	—
Barium oxide	BaO	153	76·5	$BaH_2O_2.8Aq.$ 1 to 20	$\frac{E}{3·15}$
Calcium oxide	CaO	56	28	Saturated solution	$\frac{E}{20}$
Ammonium sulphide	Am_2S	68	34	10 % NH_3	5·54 E ?
„	„	„	„	—	—
Sodium sulphide	Na_2S	78	39	9 % Na_2O	3·28 E
„	„	„	„	—	—
Potassium cyanide	KCy	65	65	—	—
Potassium sulphate	K_2SO_4	174	87	1 to 12	0·96 E
Potassium iodide	KI	166	166	—	—
„	„	„	„	—	—
Potassium chromate	K_2CrO_4	194·5	97·25	—	—
Potassium metantimoniate	$KSbO_3$	209	209	Saturated solution	$\frac{E}{68}$
Potassium ferrocyanide	K_4FeCy_6	368	92	1 to 12	0·79 E
Potassium ferricyanide	$K_6Fe_2Cy_{12}$	658	109·7	—	—
Potassium sulphocyanate	KCyS	97	97	1 to 10	1·03 E
Sodium carbonate	Na_2CO_3	105	53	2·7 to 5	3·77 E
„	„	„	„	—	—
Hydrogen disodium-phosphate	HNa_2PO_4	142	47·3	1 to 10	0·84 E
Sodium acetate	$NaC_2H_3O_2$	82	82	1 to 10	0·74 E
Sodium sulphite	Na_2SO_3	126	63	—	—
Sodium thiosulphate	$Na_2S_2O_3$	158	79	—	—
Sodium hypochlorite	NaClO	74·5	74·5	—	—
Ammonium acetate	$AmC_2H_3O_2$	77	77	—	—
„	„	„	„	—	—
Ammonium oxalate	$Am_2C_2O_4$	124	62	1 to 24	0·59 E
Hydrogen di-ammonium phosphate	HAm_2PO_4	132	44	—	—
Ammonium carbonate	Am_2CO_3	96	48	1 sesqui to 5 + NH_3	5·08 E
„	„	„	„	—	—

Column 6. Equivalent System. Grms. per litre.	Equivalent System. Strength.	7. Symbol of substance taken.	7. Method of Preparing Reagent.	8. Strength of Reagent as found by trial.
35	E	—	200 cc. of 5 E ammonium hydrate diluted to 1 litre.	E
—	$\frac{E}{3}$	BaH_2O_2, 8Aq	52·5 grms. dissolved and diluted to 1 litre. Note (15).	—
—	$\frac{E}{20}$	—	Water at 15·5° C. saturated with calcium hydrate.	$\frac{97\ E}{2000}$
—	5 E	—	Saturate 600 cc. of 5 E ammonium hydrate with H_2S in a corked flask, and then add 400 cc. of 5 E ammonium hydrate.	5 E
34	E	—	200 cc. of 5 E ammonium sulphide diluted to 1 litre.	E
—	5 E	—	Dissolve 200 grms. of sodium hydrate in 800 cc. water, saturate one half with H_2S, then add the other half and dilute to 1 litre.	5 E
39	E	—	200 cc. of 5 E sodium sulphide diluted to 1 litre.	E
65	E	KCy	65 grms. crystal dissolved and diluted to 1 litre.	—
87	E	K_2SO_4	87 grms. dissolved and diluted to 1 litre.	—
166	E	KI	166 „ „ „ Note (18).	E
—	$\frac{E}{5}$	„	33·2 „ „ „ Note (18).	$\frac{E}{5}$
97·25	E	K_2CrO_4	97·25 „ „ „	E
3·07	$\frac{E}{68}$	$K_2Sb_2O_6$, 7Aq	Saturated solution at 15·5° C.	—
92	E	K_4FeCy_6, 3Aq	105·5 grms. dissolved and diluted to 1 litre.	E
109·7	E	$K_6Fe_2Cy_{12}$	109·7 „ „ „	E
97	E	KCyS	97 „ „ „	E
—	3 E	$Na_2CO_3$10Aq	429 grms. dissolved and diluted to 1 litre.	2·94 E
53	E	„	143 „ „ „	0·98 E
47·3	$\frac{2\ E}{3}$ or E	HNa_2PO_4, 12Aq	119·3 „ „ „	E
—	4 E	$NaC_2H_3O_2$, 3Aq	544 „ „ „	3·96 E
—	2 E —	Na_2SO_3, 7Aq	252 „ „ „	—
79	$\frac{E}{2}$ or E	$Na_2S_2O_3$, 5Aq	124 „ „ „	—
—	—	—		—
—	5 E	—	294 cc. of 17 E acetic acid, neutralised with strong ammonium hydrate and diluted to 1 litre.	5 E
77	E	—	200 cc. of 5 E ammonium acetate diluted to 1 litre.	E
37·2	$\frac{3\ E}{5}$	$Am_2C_2O_4$, Aq	42·6 grms. dissolved and diluted to 1 litre. Note (16).	$\frac{3\ E}{5}$
44	$\frac{2\ E}{3}$ or E	HAm_2PO_4	44 grms. dissolved and diluted to 1 litre.	E
—	5 E	—	196·7 grms. of ammonium sesquicarbonate dissolved in 333·3 cc. of 5 E ammonium hydrate and diluted to 1 litre.	—
48	E	—	200 cc. of 5 E ammonium carbonate solution diluted to 1 litre.	—

Column 1.	2.	3.	4.	5.	
				Fresenius's System.	
				Strength of Reagent.	
Name of Reagent.	Symbol.	Molecular weight.	Equivalent weight.	As stated.	Calculated in equivalents.
Hydrogen ammonium carbonate	$HAmCO_3$	79		—	—
Ammonium chloride ...	$AmCl$	53·5	53·5	1 to 8	2·34 E
„ ...	„	„	„	—	—
Ammonium sulphate ...	Am_2SO_4	132	66	—	—
Barium chloride	$BaCl_2$	208	104	1 to 10	0·82 E
Barium nitrate	BaN_2O_6	261	130·5	1 to 15	0·51 E
Barium carbonate	$BaCO_3$	197	98·5	—	—
Strontium sulphate ...	$SrSO_4$	183·5	91·75	—	—
Calcium chloride	$CaCl_2$	111	55·5	1 cryst. to 5	1·83 E
Calcium sulphate	$CaSO_4$	136	68	Saturated solution	$\frac{E}{30}$
Magnesium chloride ...	$MgCl_2$	95	47·5	—	—
Magnesium sulphate ...	$MgSO_4$	120	60	1 to 10	0·81 E
Ferrous sulphate	$FeSO_4$	152	76	—	—
Ferric chloride	Fe_2Cl_6	325	54·17	—	—
Plumbic acetate	$PbC_4H_6O_4$	325	162·5	1 to 10	0·53 E
Plumbic nitrate	PbN_2O_6	331	165·5	—	—
Argentic nitrate	$AgNO_3$	170	170	1 to 20	0·30 E
„	„	„	„	—	—
Argentic sulphate	Ag_2SO_4	312	156	—	—
Mercurous nitrate	$Hg_2N_2O_6$	524	262	—	—
Mercuric chloride	$HgCl_2$	271	135·5	1 to 16	0·46 E
Cupric sulphate	$CuSO_4$	159·5	79·75	1 to 10	0·80 E
Cupric chloride	$CuCl_2$	134·5	67·25	—	—
Stannous chloride	$SnCl_2$	189	94·5	—	—
Auric chloride	$AuCl_3$	303·1	101	1 to 30	0·33 E
Platinic chloride	$PtCl_4$	339·1	84·8	—	0·77 E
Magnesia mixture (for phosphoric acid)	—	—	—	—	—

Column 6.		7.		8.
Equivalent System.				
Grms. per litre.	Strength.	Symbol of substance taken.	Method of Preparing Reagent.	Strength of Reagent as found by trial.
—	$\frac{3E}{2}$ or 3 E	—	A saturated solution made by passing excess of carbon dioxide into 3 E ammonium hydrate.	—
—	5 E	AmCl	267·5 grms. dissolved and diluted to 1 litre.	5 E
53·5	E	,,	53·5 ,, ,, ,,	E
66	E	Am_2SO_4	66 ,, ,, ,,	E
104	E	$BaCl_2$, 2Aq	122 ,, ,, ,,	E
65·25	$\frac{E}{2}$	BaN_2O_6	65·25 ,, ,, ,, Note (17).	$\frac{E}{2}$
—	2 E	$BaCO_3$	197 grms. freshly precipitated, suspended in water and diluted to 1 litre.	—
0·153	$\frac{E}{600}$ —	$SrSO_4$	Water at 15·5° C. saturated with precipitated strontium sulphate.	—
55·5	E	$CaCl_2$, 6Aq	109·5 grms. dissolved and diluted to 1 litre.	0·97 E
2·27	$\frac{E}{30}$	$CaSO_4$, 2Aq	Water at 15·5° C. saturated with precipitated calcium sulphate.	—
47·5	E	$MgCl_2$, 6Aq	101·5 grms. dissolved and diluted to 1 litre.	0·98 E
60	E	$MgSO_4$, 7Aq	123 grms. dissolved and diluted to 1 litre.	E
76	E	$FeSO_4$, 7Aq	139 ,, ,, ,,	0·97 E
54·17	E	—	18·67 grms. of iron, as $Fe_2H_6O_6$, dissolved in 200 cc. of 5 E hydrochloric acid and diluted to 1 litre.	—
162·5	E	$PbC_4H_6O_4$, 3Aq	189·5 grms. dissolved and diluted to 1 litre.	E
165·5	E	PbN_2O_6	165·5 ,, ,, ,,	0·98 E
170	E	$AgNO_3$	170 ,, ,, ,, Note (18).	E
—	$\frac{E}{5}$	,,	34 ,, ,, ,, Note (18).	$\frac{E}{5}$
7·8	$\frac{E}{20}$	Ag_2SO_4	Water at 15·5° C. saturated with freshly precipitated argentic sulphate.	$\frac{E}{21}$
52·4 +	$\frac{E}{5}$ +	$Hg_2N_2O_6$, 2Aq	56 grms. dissolved in 40 cc. of 5 E nitric acid and diluted to 1 litre, a little mercury being placed in a bottle.	$\frac{E}{5}$
54·2	$\frac{2E}{5}$	$HgCl_2$	54·2 grms. dissolved and diluted to 1 litre.	$\frac{2E}{5}$
79·75	E	$CuSO_4$, 5Aq	124·75 ,, ,, ,,	E
67·25	E	$CuCl_2$, 2Aq	85·25 ,, ,, ,,	0·95 E
94·5 +	E +	$SnCl_2$, 2Aq	112·5 grms. dissolved in 200 cc. of 5 E hydrochloric acid and diluted to 1 litre, a little tin being placed in bottle.	0·96 E
20·2	$\frac{E}{5}$	—	13·1 grms. of metallic gold, converted into auric chloride, dissolved and diluted to 1 litre. Note (18).	—
84·8	E	—	49·3 grms. of metallic platinum, converted into platinic chloride, dissolved and diluted to 1 litre. Note (18).	—
—	E	—	Dissolve 68 grms. $MgCl_2$, 6 Aq in about 500 cc. of water, add 165 grms. AmCl, then 300 cc. of 5 E ammonium hydrate, and dilute to 1 litre.	—

(7) Acetic acid, anhydrous, sp. gr. 1·0635, containing 100 per cent., by calculation gives 17·7 E.

Acetic acid, sp. gr. 1·0598, solid at 50° F., as supplied, titrated, gave 16·9 E.

(8) Oxalic acid. A $\frac{3\,E}{2}$ solution does not crystallise out at 15° C., but does so at 10° C.

(9) Bromine, sp. gr. 2·966, by calculation gives 37·08 E.

(10) Hydrogen peroxide, 20 volumes, by calculation gives 3·6 E.

(11) Hydrogen peroxide, 10 volumes, by calculation gives 1·8 E.

(12) Potassium hydrate, about one-tenth more than the quantity given in the table is required, owing to most samples containing water.

(13) Sodium hydrate prepared from sodium is employed.

(14) Ammonium hydrate, sp. gr. 0·88, containing 38 per cent., by calculation gives 19·7 E.

Ammonium hydrate, sp. gr. 0·88, as supplied, three samples titrated gave 18·0 E, 16·2 E, and 18·9 E.

(15) Barium hydrate. The strength of a *saturated* solution varies considerably with the temperature.

(16) Ammonium oxalate. A stronger solution than $\frac{3\,E}{5}$ crystallises out at the ordinary temperature.

(17) Barium nitrate is not sufficiently soluble to make an E solution.

(18) Where reagents are expensive an $\frac{E}{5}$ or $\frac{E}{10}$ solution may be made for qualitative tests.

It will be seen from the Table that a large number of the reagents are made of E strength. Experience, however, has shown that there are cases in which it is necessary to employ certain reagents, and especially the acids, stronger than this; consequently, a number of 5 E reagents have been introduced, 5 being a convenient figure for calculations on the decimal system. These are more accurately made, and keep more constant in strength than the concentrated reagents, which are liable to a variation of about one equivalent.

About three-fourths of the reagents given in the Table may be kept in well-stoppered bottles for a considerable time without appreciable change. In the few remaining cases where the variation in strength is considerable, the unwelcome fact is expressed in column 6 of the preceding Table and on the reagent bottles in the following manner:—

Where it is known that the reagent has been made the standard strength, but becomes weaker on keeping, it may be expressed by the sign – placed after the symbol E, thus:—4 E — Sulphurous Acid. If, on the other hand, there is an increase of strength, as in the case of the acid solution of stannous chloride kept in contact with metallic tin, it may be expressed thus:—E + Stannous Chloride. The analyst is thus forewarned as to any irregularity which may occur in the strength of such reagents.

The practical application of these multiple and fractional equivalents is well illustrated by the chlorine and bromine reagents given in the table. Pure liquid bromine is shown as 37 E; a saturated solution of bromine in water as $\frac{E}{2}$; and a saturated solution of chlorine in water $\frac{E}{5}$. Now if we know that 1 cc. of liquid bromine (37 E) is required to effect a definite amount of oxidation in a solution, we see at a glance by means of these equivalents that it would require 74 cc. of $\frac{E}{2}$ bromine water or 185 cc. of $\frac{E}{5}$ chlorine water to effect the same *quantitative* result.

It may here be pointed out that a few reagents require a double index of their strength, *e.g.*, E or $\frac{2\,E}{3}$ hydrogen disodium phosphate.* This reagent behaves as an E solution if employed to precipitate iron as phosphate, but only as $\frac{2\,E}{3}$ when employed to precipitate magnesium as ammonium magnesium phosphate owing to ammonium taking part in the reaction. A reference to the chemical equations representing the reactions will illustrate this:—

---

* On this account the second or third label previously given is preferred.

$$\underbrace{Fe_2Cl_6}_{\text{6 equivs.}} + \underbrace{2\,HNa_2PO_4}_{\text{6 equivalents.}} + 2\,Na\bar{A} = \underbrace{2\,FePO_4 + 6\,NaCl + 2\,H\bar{A}}_{\text{6 equivs. of Fe precipitated.}}.$$

$$\underbrace{2\,MgCl_2}_{\text{4 equivs.}} + \underbrace{2\,HNa_2PO_4}_{\text{6 equivalents.}} + 2\,AmHO = \underbrace{2\,AmMgPO_4 + 4\,NaCl + 2\,H_2O}_{\text{4 equivs. of Mg precipitated.}}.$$

Having thus briefly described the proposed system of reagents, we will proceed to notice some of the advantages attending its employment in practical and analytical chemistry:—

1. It affords the most convenient method of expressing the *proportionate* strength or precipitating power of each reagent, and one which may be plainly indicated on each bottle.

2. It indicates as near as can be practically attained the *actual* strength of each reagent.

Thus:—36 E sulphuric acid contains 36 m.grm. equivalents in 1 cc., and knowing this we can readily calculate the quantity of any constituent, *e.g.*, the equivalent weight of sulphuric acid being 49, we have 49 × 36 = 1,764 m.grms. = 1·764 grms. of sulphuric acid present in 1 cc. Similarly, each cc. contains 1 × 36 = 36 m.grms. = 0·036 grm. of hydrogen, 16 × 36 = 576 m.grms. = 0·576 grm. of sulphur, and 32 × 36 = 1,152 m.grms. = 1·152 grms. of oxygen.

3. By observing the quantity of a reagent employed in an analysis, we can calculate the quantity of by-product therefrom, a point of considerable importance in deciding upon the dilution of our solutions.

For example:—If, during a quantitative analysis, 10 cc. of 36 E sulphuric acid is added and afterwards neutralized by sodium carbonate, we observe that 10 × 36 equivalents of sodium sulphate will be produced. Now, since the equivalent of sodium sulphate is 71, we have 71 × 36 ×

·10 = 25,560 m.grms. = 25·56 grms. as the quantity of anhydrous sodium sulphate formed by the reaction. Knowing this, we are forewarned to dilute sufficiently before proceeding with the analysis.

4. In qualitative analysis we are able to form a better judgment of the amount of substance present than when using the reagents of indefinite strength hitherto employed.

5. The strength of reagents employed in chemical investigations may be most conveniently expressed on this system.

For example :—

Nitric acid sp. gr. 1·4268 at 15·5° C. is *exactly* 16 E in strength.
" " 1·1656 " " 5 E "
" " 1·0345 " " E "

Now it is evident that the latter mode of expression is far simpler, and gives a much more accurate idea of the strength of the acid than does its specific gravity. These equivalent numbers would be a valuable addition to sp. gr. tables.

6. Reagents may be most readily diluted to the various strengths required in chemical analysis.

For example :—Nitric acid sp. gr. 1·42 = 16 E approximately may be readily obtained by distillation. To make an acid of E strength, it is simply necessary to dilute 1 cc. of 16 E acid with water to the volume of 16 cc. To make 2 E acid, dilute 2 cc. of 16 E acid to 16 cc. ; or to make 15 E acid, take 15 cc. of 16 E acid and dilute to 16 cc., and so on. It is not usually necessary to take changes of temperature into consideration, except in the case of strong sulphuric acid.

7. This system affords the simplest method of calculating the theoretical quantity of reagent required to effect any chemical change.

For example :—If we wish to find the quantity of reagent necessary to precipitate 1 grm. of calcium from its solution ; we already know that 1 cc. of E reagent, say E ammonium carbonate, will precipitate 20 m.grms. (1 m.grm.

equivalent); consequently, 50 cc. of E reagent will be required to precipitate 1 grm. If preferred, 10 cc. of 5 E ammonium carbonate may be used instead. In like manner we can calculate the quantity of acid theoretically required to dissolve a given quantity of metal or other substance. Thus, 1 grm. of zinc, containing 30·8 m.grms. equivalents, will theoretically require 30·8 cc. of E acid or 6·16 cc. of 5 E acid to effect its solution. In the same way it may be shown that while to dissolve or precipitate 1 grm. of thallium theoretically requires only 4·9 *cc. of E* reagent, to dissolve or precipitate 1 grm. of beryllium would require 217·4 *cc. of E reagent.*

The following table gives the number* of m.grm. equivalents contained in 1 grm. of each of the more common elementary substances. This enables us to find at a glance the number of cc. of E reagent theoretically required to effect the solution or precipitation of 1 grm. of each substance.

[illegible]	Atomic Weight.	Equivalent Weight.	M.grm. equivs. per grm., or cc. of E.
[illegible]	[illegible]	[illegible]	111·1
[illegible]	[illegible]	[illegible]	25
[illegible]	[illegible]	24	41·7
[illegible]	[illegible]	25	40
[illegible]	[illegible]	15	66·7
[illegible]	[illegible]	68·5	14·6
[illegible]	[illegible]	[illegible]	217·4
[illegible]	[illegible]	[illegible]	14·3
[illegible]	[illegible]	[illegible]	25·3
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	12·5
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]

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* [illegible] weight [illegible] × 1000; con- [illegible] substances and [illegible]

Symbol of Element.	Atomic weight.	Equivalent weight.	M.grm. equivs. per grm., or cc. of E.
Cr'''	52·5	17·5	57·1
Cr^{vi}	,,	8·75	114·3
Co''	59	29·5	33·9
Co'''	,,	19·67	50·8
Cu'	63·5	63·5	15·7
Cu''	,,	31·75	31·5
F'	19	19	52·6
Au'	196·6	196·6	5·1
Au'''	,,	65·53	15·3
H'	1	1	1000·0
I'	127	127	7·9
Fe''	56	28	35·7
Fe'''	,,	18·67	53·6
Pb''	207	103·5	9·7
Li'	7	7	142·9
Mg''	24	12	83·3
Mn''	55	27·5	36·4
Mn^{iv}	,,	13·75	72·7
Mn^{vi}	,,	9·17	109·1
Hg'	200	200	5
Hg''	,,	100	10
Ni''	59	29·5	33·9
Ni'''	,,	19·67	50·8
P'''	31	10·33	96·8
P^{v}	,,	6·2	161·3
Pt''	194·381	97·19	10·3
Pt^{iv}	,,	48·59	20·6
K'	39·1	39·1	25·6
Si^{iv}	28	7	142·9
Ag'	108	108	9·3
Na'	23	23	43·5
Sr''	87·5	43·75	22·9
S''	32	16	62·5
S^{iv}	,,	8	125
S^{vi}	,,	5·33	187·6
Sn''	118	59	16·9
Sn^{iv}	,,	29·5	33·9
Tl'	204	204	4·9
Tl'''	,,	68	14·7
Ti''	50	25	40
Ti^{iv}	,,	12·5	80
Zn''	65	32·5	30·8

equivalent); consequently, 50 cc. of E reagent will be required to precipitate 1 grm. If preferred, 10 cc. of 5 E ammonium carbonate may be used instead. In like manner we can calculate the quantity of acid theoretically required to *dissolve* a given quantity of metal or other substance. Thus, 1 grm. of zinc, containing 30·8 m.grms. equivalents, will theoretically require 30·8 cc. of E acid or 6·16 cc. of 5 E acid to effect its solution. In the same way it may be shown that while to dissolve or precipitate 1 grm. of thallium theoretically requires only 4.9 *cc. of E reagent*, to dissolve or precipitate 1 grm. of beryllium would require 217·4 *cc. of E reagent.*

The following table gives the number* of m.grm. equivalents contained in 1 grm. of each of the more common elementary substances. This enables us to find at a glance the number of cc. of E. reagent theoretically required to effect the solution or precipitation of 1 grm. of each substance.

Symbol of Element.	Atomic Weight.	Equivalent Weight.	M.grm. equivs. per grm., or cc. of E.
Al'''	27	9	111·1
Sb'''	120	40	25
Sb^{v}	,,	24	41·7
As'''	75	25	40
As^{v}	,,	15	66·7
Ba''	137	68·5	14·6
Be''	9·2	4·6	217·4
Bi'''	210	70	14·3
Bi^{v}	,,	42	23·8
B'''	11	3·67	272·5
Br'	80	80	12·5
Cd''	112	56	17·9
Ca''	40	20	50
C''	12	6	166·7
C^{iv}	,,	3	333·3
Cl'	35·5	35·5	28·2

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* This number is the reciprocal of the equivalent weight × 1,000; consequently a table of reciprocals would be applicable for all substances, and might be used for this purpose.

Symbol of Element.	Atomic weight.	Equivalent weight.	M.grm. equivs. per grm., or cc. of E.
Cr'''	52·5	17·5	57·1
Cr^{vi}	,,	8·75	114·3
Co''	59	29·5	33·9
Co'''	,,	19·67	50·8
Cu'	63·5	63·5	15·7
Cu''	,,	31·75	31·5
F'	19	19	52·6
Au'	196·6	196·6	5·1
Au'''	,,	65·53	15·3
H'	1	1	1000·0
I'	127	127	7·9
Fe''	56	28	35·7
Fe'''	,,	18·67	53·6
Pb''	207	103·5	9·7
Li'	7	7	142·9
Mg''	24	12	83·3
Mn''	55	27·5	36·4
Mn^{iv}	,,	13·75	72·7
Mn^{vi}	,,	9·17	109·1
Hg'	200	200	5
Hg''	,,	100	10
Ni''	59	29·5	33·9
Ni'''	,,	19·67	50·8
P'''	31	10·33	96·8
P^{v}	,,	6·2	161·3
Pt''	194·381	97·19	10·3
Pt^{iv}	,,	48·59	20·6
K'	39·1	39·1	25·6
Si^{iv}	28	7	142·9
Ag'	108	108	9·3
Na'	23	23	43·5
Sr''	87·5	43·75	22·9
S''	32	16	62·5
S^{iv}	,,	8	125
S^{vi}	,,	5·33	187·6
Sn''	118	59	16·9
Sn^{iv}	,,	29·5	33·9
Tl'	204	204	4·9
Tl'''	,,	68	14·7
Ti''	50	25	40
Ti^{iv}	,,	12·5	80
Zn''	65	32·5	30·8

Should a secondary reaction occur, as for instance in the action of copper upon nitric acid, the quantity of reagent required may be deduced from the equation representing the chemical change, as follows :—

$$\underbrace{3\,Cu}_{\text{6 equivalents.}} + \underbrace{8\,HNO_3}_{\text{8 equivalents.}} = 3\,CuN_2O_6 + 2\,NO + 4\,H_2O.$$

Here we see that 6 equivalents of copper require 8 equivalents of nitric acid, or one-third more than the normal quantity; therefore 1⅓ times the quantity of acid, as obtained from the table, will be required.

8. The system further affords a simple method of calculating the quantity of gas given off in chemical reactions.

Since 1 cc. of an E reagent, in acting upon excess of metal, will liberate a m.grm. equivalent of hydrogen = 11·16 cc., the volume given off by x cc. of any E reagent will be x 11·16 cc. Conversely, we may calculate the quantity of acid required to produce a given volume of hydrogen.

9. In working with this system we have the most convenient method of expressing the precise acidity, &c., of chemical solutions intended for accurate titration, precipitation, &c.

For example :—In Margueritte's method of estimating iron volumetrically by means of *standard* $\frac{E}{10}$ potassium permanganate, I recommend that the solution for titration be acidified to E or $\frac{E}{2}$ sulphuric acid, thus expressing the acidity at which it is preferred the reaction should take place.

An example of the application of these reagents to the *separation of metals* may be given. It is known that a solution of zinc chloride containing 1 grm. of zinc. in 100 cc. requires the presence of an appreciable quantity of free hydrochloric acid to prevent the precipitation of the *zinc* by hydrogen sulphide. It is also known that *lead* is not completely precipitated from its solutions by hydrogen sulphide if more than 2·5 per cent. of free hydrochloric acid be present. The question arises how to acidify the solution so as to allow of the complete precipitation of the *lead* without the co-precipitation of the *zinc.* I have found that *lead* can be completely precipitated by hydrogen sulphide from an $\frac{E}{2}$ hydrochloric acid solution without perceptible co-precipitation of *zinc,* and now by acidifying the solution to $\frac{E}{2}$ hydrochloric acid, I always secure the most favourable conditions for the separation of these metals.

As illustrating the application of this system to *precipitation,* I may mention the determination of *lead as sulphate.* It is well known that lead sulphate is soluble in strong sulphuric acid, and also in pure water, but that in dilute sulphuric acid it is practically insoluble. The strength of sulphuric acid corresponding to the least solubility does not appear to have been recorded. I have, however, found that lead sulphate is least soluble in *E sulphuric acid,* and now always adopt this strength in my analytical work.

As a final illustration we will take the precipitation of phosphoric acid by magnesia mixture.

David Lindo, in *Chemical News,* vol. xlviii., p. 217, has given the conditions under which he considers this precipitation can be best effected. I have calculated the quantities of reagent used in terms of E, and the conditions of precipitation may be stated as follows :—

1st. Solution to be neutral or slightly ammoniacal.

2nd. Dilution of P_2O_5 to be from $\frac{E}{5}$ to $\frac{E}{20}$, or about 1 grm. in from 200 to 800 cc.

3rd. Dilution of AmCl to be from E to $\frac{E}{2}$.

4th. After precipitate has settled, add ¼ bulk of 5 E ammonium hydrate.

5th. Wash with $\frac{3\,E}{2}$ ammonium hydrate.

6th. Magnesia mixture up to $\frac{E}{3}$ in excess does not vitiate result.

7th. Magnesia mixture counteracts solvent action of ammonium chloride.

Preparation of *Standard* Equivalent Solutions.—In volumetric analysis, where the measure of a certain substance is to be obtained from the volume of a certain reagent required to neutralize or precipitate it, it is essential that its value should be *accurately* known, so that a definite volume of it will be equivalent to, or a measure of, any substance which it is desired to determine. The following method used for preparation of standard solutions in alkalimetry, acidimetry, &c., will suffice to illustrate how *Standard* equivalent solutions are prepared.

Preparation of Standard E Sodic Carbonate.—Since sodic carbonate can be obtained in a pure state, and any moisture it may contain be driven off by gentle ignition, it can be used as a basis for the standardization of all acid and alkaline solutions. It is prepared by dissolving 53 grms. of pure anhydrous sodic carbonate in water and diluting to 1,000 cc. at 15° C. About 56 grms. of the carbonate are weighed out into a platinum dish, and gently ignited to a dull red heat until all moisture has been eliminated; it is cooled in a desiccator, and the 53 grms. quickly weighed from it.

Preparation of Standard E Sulphuric Acid.—Standard E sulphuric acid should contain 49 grms. real H_2SO_4 in a litre of water.

The following will illustrate how this is obtained:—29 cc. of strong sulphuric acid of sp. gr. 1·84 was dissolved in water, and diluted to 1,000 cc. at 15° C. 30 cc. of standard E Na_2CO_3 was run from a burette into a 100 cc. beaker, and two drops of methyl orange (1 in 1,000) added, and diluted to 50 cc. A burette was filled with the H_2SO_4 prepared as above, and allowed to gradually run into the yellow-tinted alkaline solution, stirring, until the last drop added produced a pink coloration, when 28 cc. were required instead of 30 cc. Now it is necessary to add water to the acid, since it is too strong, to an extent that will make it exactly E, and every 28 cc. of the acid will want diluting to 30 cc. There was 910 cc. of acid left; therefore $\frac{910}{28} = 32·5$ times; and $32·5 \times 2 = 65$ cc. more water to be added; on adding this quantity, and again titrating as above, 30 cc. of alkali required exactly 30 cc. of acid, and therefore, the solutions were chemically equivalent to each other.

Preparation of Standard E Sodic Hydrate.—A Standard E solution of sodic hydrate should contain 40 grms. of NaHO in a litre, but since pure sodic hydrate always contains some water, a quantity more than this must be taken. 42 grms. of pure sodic hydrate prepared from sodium was dissolved in water and diluted to 1,000 cc. at 15° C., and on titrating 30 cc. with the above standard E acid, exactly 30 cc. were required for neutralization; but often it requires more acid than this, then it must be diluted until it is of the right strength, in a similar manner to that adopted for the dilution of the sulphuric acid.

PART I.

METALS, ALLOYS, ETC.

IRON AND STEEL.

Iron, in its various forms, such as cast iron, malleable iron, and steel, is the most serviceable and largely used of all the metals. The ores from which it is extracted are extensively distributed through the crust of the earth, and the metal is easily reduced to "cast iron" by mixing the ore with certain proportions of coke or coal and limestone and smelting in a blast-furnace. The principal ores from which iron is obtained are—*Magnetic Iron Ore*, occurring largely in Sweden, Norway, Russia, and North America, and containing, when pure, some 72 per cent. of iron combined with oxygen; *Red Hæmatite*, a peroxide of iron, found in Lancashire, Cumberland, and Glamorganshire, containing from 40 to 70 per cent. of iron; *Brown Hæmatite*, an hydrated oxide of iron, found in France, Cumberland, and Durham, and containing 40 to 60 per cent. of iron.

Specular, or Micaceous Ore, is a crystalline peroxide of iron, found in Spain, Nova Scotia, and the Island of Elba; *Spathic Iron Ore* is a carbonate of iron, containing 30 to 48 per cent. of iron, found in Somersetshire and Durham, and also in Corinthia and Styria; *Clay Ironstone, or Argillaceous Iron Ore*, is a carbonate of iron associated more or less with clay; it is the source of much of the iron produced in this country; it occurs largely in South Wales, Yorkshire, Staffordshire, and

Derbyshire, and also in Belgium and Silesia; *Blackband Iron Ore* is similar in constitution to clay ironstone, but in addition to the other substances it contains more or less coaly or bituminous matter; the amount of iron present varies from 20 to 40 per cent.

The iron-smelting industries are largely carried on in Great Britain, Germany, Belgium, France, and the United States. Iron, immediately reduced from its ores, is termed cast iron and pig iron, which may be white or grey, qualities depending upon the condition and proportion of carbon present; the grey variety containing this element in the uncombined form as graphite, while in the white it is chemically combined with the iron, producing a metal so hard and brittle that a file has scarcely any influence upon it. The grey iron is soft, and may be easily filed and turned in a lathe. The conditions necessary to the formation of white and grey iron in the blast-furnace depend upon the amount of fuel used and the temperature to which the metal has been raised; a high temperature is conducive to the production of grey iron, while a low temperature produces white iron. The maximum amount of carbon which is taken up by iron in the blast-furnace appears to be about 4½ per cent. Pig iron contains, besides carbon, notable quantities of silicon, together with sulphur, phosphorus, manganese, and small quantities of copper, arsenic, aluminium, and titanium. Small quantities of these elements play a very important part in the physical properties of the metal.

Wrought Iron.—This variety of iron is produced from cast iron by a process of oxidation, the iron being melted in a furnace with slags rich in oxides of iron, fresh surfaces of the metal being continually exposed by the action of a powerful oxidising flame with a rabbler, by which means the greater portion of carbon, silicon, sulphur, and phosphorus are eliminated. The physical properties of wrought iron are totally different from those of the original cast iron; it is malleable, ductile, fibrous, and weldable. The ultimate tensile strength of good wrought iron is about 25 lbs. per square inch. The manufacture of wrought iron by the puddling process is now not nearly so extensive as formerly, since steel, which is made so cheaply by the Siemens and Bessemer processes, is replacing it.

Steel.—Steel differs from wrought iron in containing more combined carbon, being virtually an alloy of iron and a small quantity of carbon. There is no hard and fast line, however, which can be said to definitely divide the two varieties. Speaking generally, the presence of from 0·2 to 1·5 per cent. of carbon constitutes steel, provided the presence of other elements is not so high as to interfere with its influence on the metal. The tensile strength of steel depends upon the amount of carbon present and also the rate of cooling; it varies from 30 to 50 tons per sq. in.

There are several processes by which steel can be cheaply produced from wrought iron and pig iron. In the manufacture of fine steel for cutlery, &c., the cementation process is used, where the requisite quantity of carbon is given to wrought iron to convert it into steel, by keeping bars of the former surrounded with charcoal at a red heat, in a specially constructed furnace, for a definite length of time, depending upon the quality of steel required; the longer the bars are left in the furnace the more steely do they become. The process is a rather long one, occupying from five days to a fortnight in completion.

The theory of the absorption of carbon by this process is not thoroughly understood. The general idea is, that the small quantity of oxygen in the air surrounding the metal combines with that contained in the charcoal, forming carbonic oxide gas. Now, carbonic oxide is absorbed by iron at a red heat, and under the conditions of the furnace is dissociated into carbon and oxygen, the former being retained in the metal while the latter creates and combines with more carbon, forming carbonic oxide, which is again absorbed by the metal and dissociated as before, the same oxygen being used over and over again as a carrier of carbon.

Steel thus produced is not thoroughly homogeneous, and in order to get a thorough distribution of carbon the bars are cut into small pieces and melted in narrow crucibles, made of fire-clay and plumbago, about two feet high, and then cast into rectangular or octagonal moulds which have been previously heated. Great care is required in the melting of the steel; if the metal is heated to too high a temperature before pouring, it will *teem* fiery, or boil, owing to escape of gases, producing an ingot which will be rendered useless by blow-holes. Experience is required, therefore, to know the right temperature.

Large quantities of soft steel are now being made by the regenerative open-hearth process of Sir William Siemens. Steel may be produced in this furnace by three different methods:— (1) By melting pig iron and oxide of iron (hematite iron ore, &c.), by which means silicon, sulphur, and phosphorus are eliminated, and the carbon reduced to the proper point; (2) the melting together of scrap iron and pig iron in certain proportions; (3) by a combination of these two processes, which is known as the Siemens-Martin process.

Bessemer Process.—In this process, which may be considered the greatest metallurgical discovery of the age, vast quantities of steel are produced for the manufacture of rails, tyres, tin bars, &c. It consists in the forcing of air through molten pig iron, contained in a globular vessel lined with a highly silicious material, generally ganister, and termed a *converter*, by which means the carbon and silicon are oxidized, the carbon burning off as carbonic oxide, producing a very large flame, and the silicon oxidized to silica (SiO_2). The right quantity of carbon is added, after the metal is purified, by running in a certain proportion of a molten triple compound of carbon, manganese, and iron, termed Spiegeleisen. Phosphorus is not eliminated by this process, which therefore is only applicable to pig iron tolerably free from that element.

This difficulty, however, is overcome by lining the converter with a basic material according to what is known as the Gilchrist-Thomas, or "basic" process, and enormous quantities of phosphoric pig iron are converted into steel by its means. The lining of the converter is composed of a mixture of magnesian limestone and coal-tar, consolidated by heat. The slag produced by the basic process, owing to its containing a large quantity of phosphates, is largely used as a manure.

The following are analyses of iron most suitable for the "acid" and "basic" processes:—

	Acid. Per cent.	Basic. Per cent.
Graphite	2 to 3	2·5 to 3·0
Silicon	2 to 3½	0·5 to 1·80
Sulphur	·01 to ·08	Under ·03
Phosphorus	·01 to ·08	0·8 to 3·0

Classification of Iron and Steel.—Among engineers, &c., the terms "cast iron," "wrought iron," and "steel," are used. An International Commission at Philadelphia, in 1878, adopted a classification based on the proportion and condition of carbon in the metal, as follows:—

I. *Pig iron*, with 2·3 per cent. and more of carbon, melts at a comparatively low temperature (1075° to 1275° C.), and cannot be forged.

a, *White pig iron*; all the carbon is combined with the iron; the compound is very hard, brittle, white, and is made solely for the purpose of being converted into malleable iron.

b, *Grey pig iron*; in which more or less of the carbon is present in the form of graphite. The metal is soft, tough, grey to black, and is used for conversion into malleable iron and steel, or for the production of castings.

II. *Steel* with 1·6 to 0·4 per cent. of carbon melts at 1400° C. By sudden cooling of a red-hot mass the hardness is considerably increased.

III. *Weld iron* with less than 0·4 per cent. of carbon melts at 1600° C. and above. It cannot be appreciably hardened.

The Commission further decided that:—

1. Every malleable compound of iron, containing the ordinary elements of that metal, which is obtained either by the union of pasty masses of iron, or by any process not involving fusion, and which cannot be hardened by the ordinary method, shall be called *weld iron*. This is what was formerly known as *wrought iron*.

2. Any analogous compound, which by any cause hardens, shall be called *weld steel.* This had hitherto been termed *puddled steel.*

3. Every malleable compound of iron containing the ordinary constituents of that metal, which is obtained and procured in the fused state, but which does not harden by the ordinary methods, shall be known as *ingot iron.*

4. Every compound similar to the last, but capable of hardening from any cause whatever, shall be called *ingot steel.*

The following is a description of the influence which the more important elements have upon iron :—

Iron and Carbon.—The combination of iron with carbon is the most important of all the combinations of iron. Iron immediately reduced from its ores assimilates from 1 to 4½ per cent. of this element, and may either be present in the uncombined form as graphite, or in actual chemical combination as a carbide of iron. As mentioned above, the property of the metal is totally different, even with the same proportions of carbon, unless it be present in the same form. Carbon, up to a certain point, decreases the ductility and increases the tenacity of iron. A most important function played by carbon in steel is that of tempering. The temper of steel depends upon the proportion of carbon present and the rate of cooling. The process of tempering consists in hardening the steel to its maximum by first heating it to a certain temperature, and then plunging into water or a salt solution, and then heating the steel up to a certain temperature according to the degree of hardness required, and then allowing it to cool slowly. The temperature required for a given temper is ascertained by the colour produced on the surface of the metal, due to incipient oxidation. The colour varies from a very faint yellow, through shades of yellow, brown, to dark blue. An experienced workman can thus tell when to suspend the heating.

The following table shows the temperatures and colours of steel tempered for various articles :—

Temperature F.	Colour.	Temper.
430	Very faint yellow.	Lancets.
450	Pale straw.	Razors and surgical instruments.
470	Full yellow.	Penknives.
490	Brown.	Scissors and cold chisels.
510	Brown with purple spots.	Axes, plane irons, pocket-knives.
530	Purple.	Table knives, large shears.
550	Bright blue.	Swords, watch springs.
560	Full blue.	Fine saws, augers.
600	Dark blue.	Hand and pit saws.

The *hardness* of steel containing a given quantity of carbon will depend upon the rate at which the metal was cooled, and also upon the amounts and nature of other elements present, such as silicon, sulphur, phosphorus, manganese, &c. It appears that 1·5 per cent. of carbon in a steel is the maximum amount that can be present to render it workable. Steel with 1 per cent. of carbon welds well, and makes good cold chisels.

The following are ideal percentages of carbon in steels suitable for various purposes, assuming the other elements to be present in normal quantities :—

Per cent.	
1·3 to 1·5	Razors and chilled roll tools.
1·2 to 1·4	Saw files.
1·1 to 1·2	Drills, turning tools, c.
1 to 1·1	Spindles, large turning tools, millpicks, &c.
·8 to 1·0	Shears, dies, cold sets, &c.
·3 to ·5	Tyres, rails, cannon, &c.
·2 to ·3	Boiler plates.
·15 to ·20	Crank axles.
·09 to ·13	Tin-bars.

The following analyses show that excess of silicon in presence of excess of carbon is injurious to steel used for tyres, axles, and steel plates for bridges :—

	BAD STEEL.		GOOD STEEL.	
	1 Per cent.	2 Per cent.	1 Per cent.	2 Per cent.
Combined Carbon and Graphite	·40	·53	·35	·49
Silicon	·59	·64	·05	·01
Phosphorus	·01	·03	nil	·03
Sulphur	·01	·06	,,	·03
Manganese	nil	·05	,,	·57
Copper	,,	·03	·01	·02

Much has yet to be learnt as to the state in which carbon exists in steel, and as to the exact influence exerted upon the metal by varying proportions of this element, especially in the presence or absence of other elements, and as to how far molecular change in the iron itself influences its hardness, &c. Abel has shown, from experiments made on the slow solvent action of chromic acid upon soft steel, that there exists a carbide of iron of the formula Fe_3C, and Berthier, in 1833, isolated a carbide with the formula FeC. It would appear, however, that the mode of existence of carbon in iron does not altogether explain the reason of *tempering* and *hardening*, because it is possible to obtain a soft steel by the rapid cooling of the metal containing carbon in the "hardening" form; here is doubtless a case of alteration of molecular structure of the metal itself. Osmond has advanced a theory in which it is held that there are two kinds of *iron*, the arrangement of the atoms in the molecules of which will determine the degree of hardness of the metal irrespective of the presence of carbon. Mr. Anderson has stated that "when by the agency of heat, molecular motion is raised to a pitch at which incipient fluidity is obtained, the particles of two pieces brought into contact will interpenetrate or diffuse into each other, the two pieces will unite into a homogeneous whole; we can thus grasp the full meaning of the operation of welding."

The following interesting results were obtained by Mr. Spencer, showing the influence of hardening and annealing upon the condition of carbon in steel:—

No. 1 SAMPLE.	Before Hardening. Per cent.	Hardened. Per cent.	Annealed. Per cent.
Combined Carbon (colour test)	0·89	0·58	0·98
Graphitic Carbon	0·29	trace	0·20
Total Carbon	1·18	0·58	1·18
Total Carbon by combustion	1·18	1·09	1·20
	Loss	0·51	
No. 2 SAMPLE.			
Combined Carbon (colour test)	0·80	0·66	0·79
Graphitic Carbon	0·34	0·07	0·44
Total Carbon	1·14	0·73	1·23
Total Carbon by combustion	1·21	1·10	1·19
	Loss	·37	

No. 3 Sample.	Before Hardening. Per cent.	Hardened. Per cent.	Annealed. Per cent.
Combined Carbon (colour test) .	0·38	0·22	0·38
Graphitic Carbon . . .	0·04	nil	trace
Total Carbon	0·42	0·22	0·38
Total Carbon by combustion .	0·39	0·34	0·38
Loss		·12	

Iron and Silicon.—This element when present in notable quantities produces both cold-shortness and red-shortness. Its effect, however, is influenced by the amount of other elements present, especially carbon. If a steel, for instance, contains only 0·1 per cent. of carbon, as much as 0·5 per cent. of silicon may be present without producing a brittle metal, while if in addition to the 0·5 of silicon there is present from 0·4 to 0·6 per cent. of carbon the steel will be cold-short and red-short. The presence of a small quantity of silicon in steel increases its tensile strength and lowers the limit of elasticity.

The following diagram illustrates interesting mechanical results obtained by Mr. R. A. Hadfield,* with varying quantities of silicon upon mild forged steel:—

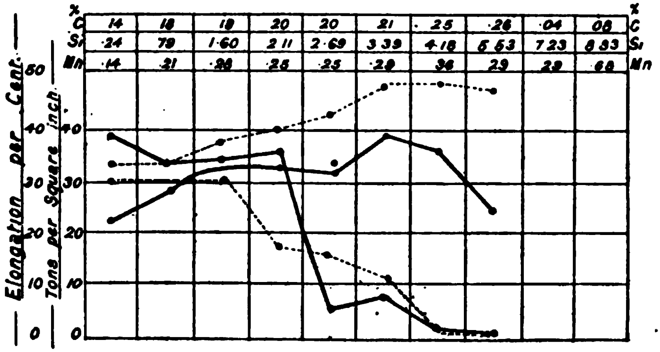

FIG. 1.

It will be seen that the upper curves indicate the results of tensile tests in tons per square inch, while the lower curves show the elongations and percentages. When the silicon present in *mild* steel exceeds about 2 per cent., its elongation rapidly diminishes.

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* *Journal of Iron and Steel Institute*, No. 1 (1889), p. 222.

Mr. Turner has studied the influence of silicon upon iron and steel, the following being results obtained by him with varying quantities of silicon in cast iron :—

Silicon Per cent.	Breaking Load. Tons per square inch.	Modulus of Elasticity.
0	10·14	25·79 millions.
0·5	12·31	28·67 ,,
1	12·72	31·18 ,,
2	15·70	23·56 ,,
2·5	14·62	25·45 ,,
3	12·23	21·15 ,,
4	11·28	15·64 ,,
5	10·16	18·72 ,,
7·5	5·34	14·75 ,,
10	4·75	13·93 ,,

It will be seen that any further addition of silicon beyond 2·5 per cent. causes a gradual deterioration in strength. Silicon plays a very important part in preventing blow-holes in iron and steel. The addition of siliceous pig iron, containing a high proportion of manganese, to molten steel, makes sound castings, the silicon preventing the formation of blow-holes owing to its oxidizing any carbonic oxide present in solution before actual solidification.

Iron and Sulphur.—The effect of sulphur upon iron and steel is to cause red-shortness, 0·1 per cent. often causing iron steel to roll badly and destroying its welding power. Manganese has a powerful effect in preventing the red-short influence of sulphur, while steel containing 0·1 per cent. of sulphur with very little manganese cannot be properly rolled, yet the presence of ·6 to 1 per cent. of manganese will make such sulphurous steel roll without a flaw.

The following are analyses of steels by Herr Wasum containing various amounts of sulphur, with remarks on their rolling properties :—

No.	Carbon.	Silicon.	Phosphorus.	Manganese.	Sulphur.	Copper.	Rolling Qualities.
1	·280	·160	·049	·634	·119	·050	Good
2	·393	·141	·065	·695	·158	·040	Good
3	·258	·136	·043	·500	·201	·076	Bad
4	·307	·075	·039	·488	·214	·057	Bad
5	·224	·089	·030	·480	·231	·066	Very bad

Iron and Manganese.—The effect of small quantities of manganese on steel is to produce sound castings and neutralize the injurious influence of sulphur; about ·6 to 1 per cent. is the usual amount present in steel rails and ·3 to ·4 per cent. in soft steels, such as tin-bars, &c. Hadfield has produced a very remarkable alloy of iron, manganese, and carbon, which he terms manganese steel, containing about 14 per cent. of manganese and about 1 per cent. of carbon. By quenching such steel from a white heat it is made very tough and strong; it rolls and forges well and is singularly free from blow-holes.

Iron and Aluminium.—The presence of 0·1 to 0·2 per cent. of aluminium hardens steel, but the metal may be rendered very soft by annealing in addition and hardening; the small quantity of aluminium materially increases the fluidity of the metal and is said to lower the melting point by some 200°.

Iron and Copper.—The effect of copper upon steel was formerly considered to render it red-short, but Herr Wasum has shown that 0·86 per cent. of copper may be present, provided the sulphur be not excessive, and yet the metal will roll perfectly sound, and Messrs. Ball and Wingham* have proved that the element increases the tensile strength of steel.

The following are analyses and effects of copper and of copper and sulphur upon the rolling qualities of steel:—

INFLUENCE OF COPPER.

	Carbon.	Silicon.	Phosphorus.	Manganese.	Sulphur.	Copper.	Rolled.
No. 1	·276	·144	·064	·778	·059	·452	Very Good.
No. 2	·233	·091	·050	·709	·060	·862	Good.

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* *Journal Iron and Steel Institute*, No. 1 (1889), p. 123.

INFLUENCE OF COPPER AND SULPHUR.

	Carbon.	Silicon.	Phosphorus.	Manganese.	Sulphur.	Copper.	Rolled.
No. 1	·311	·051	·061	·514	·107	·859	Good.
,, 2	·281	·169	·059	·594	·170	·429	Bad.
,, 3	·235	·164	·045	·468	·173	·573	Bad.
,, 4	·262	·131	·052	·655	·189	·406	Bad.

The following diagram illustrates the effect of copper upon the tensile strength of steel containing various proportions of carbon (Ball and Wingham).

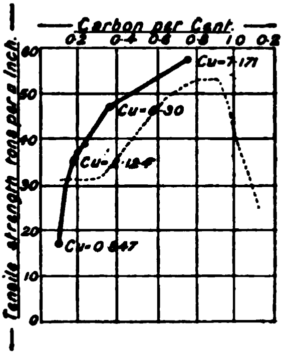

FIG. 2.

The dotted curve is that of normal carbon.

Iron and Chromium.—An appropriate addition of chromium to steel increases its tensile strength. Chrome steel is difficult to work hot, owing to the tendency of chromium to oxidize and form slag. Mr. Arnold has made experiments with steel tyres containing chromium, and the result shows that the element materially increases the tensile strength of the tyres over ordinary Bessemer steel.

The following are results obtained. The composition of an ordinary steel tyre was :—

	Per cent.
Carbon	0·280
Silicon	0·070
Manganese	1·250
Sulphur	0·080
Phosphorus	0·080

The mechanical tests of the above would give a maximum tensile strain of 37 tons per square inch; elongation, 26 per cent.; reduction of area, 47 per cent.; and fracture, grey granular with silky edges, shape convex and concave. Such a tyre, with an inside diameter of 2 ft. 8 in. and a sectional area of 11 in., would behave under the falling weight test in a manner indicated by the subjoined figures, the weight of the tup being 22 cwt.:—

Fall in Feet	2′	4′	6′	8′	10′	12′	unbroken.
Deflection in Inches	½″	¾″	1¾″	3$\frac{1}{16}$″	4$\frac{1}{16}$″	6¼″	

Steel was made, having the following composition:—

	Per cent.
Carbon	0·25
Silicon	0·03
Manganese	1·75
Sulphur	0·12
Phosphorus	0·11

The mechanical tests gave a mean maximum strain of 42·1 tons per square inch; mean elongation, 18 per cent.; mean reduction of area, 26·3 per cent. The mean results of the tests on many tyres, practically identical in composition, 2 ft. 8 in. inside diameter, and having a sectional area of 11 in., the weight of the tup being 22 cwt., were:—

Fall in Feet	2′	4′	6′	8′	10′	12′	14′
Deflection in Inches	⅛″	⅝″	1⅜″	2$\frac{3}{16}$″	3$\frac{9}{16}$″	5⅛″	6¼″

The analysis of a tyre steel required to stand a strain of at least 48 tons per square inch was:—

	Per cent.
Carbon	0·28
Chromium	0·42
Manganese	1·54

	Per cent.
Silicon	0·08
Sulphur	0·10
Phosphorus	0·09

The mechanical tests gave, maximum tensile strain, 49·8 tons per square inch; elongation, 15 per cent.; reduction of area, 26 per cent.; fracture flat and finely crystalline. A tyre of 2 ft. 8 in. diameter and of 11 in. sectional area behaved thus under the 22 cwt. falling weight:—

Fall in Feet . .	2′	4′	6′	8′	10′	12′	14′	16′	18′	20′	25′
Deflection in Inches	$\frac{1}{8}$″	$\frac{1}{2}$″	$1\frac{1}{8}$″	$1\frac{13}{16}$″	$3\frac{1}{16}$″	$4\frac{3}{8}$″	$5\frac{7}{8}$″	$7\frac{5}{8}$″	$9\frac{1}{2}$″	$11\frac{3}{16}$	broke.

A test piece planed out of the broken tyre gave the following results:—Maximum tensile strain, 47·7 tons per square inch; elongation, 3 per cent.; reduction of area, 6·4; fracture, large crystals. The molecular change set up by the shock and vibration of the falling weight is thus clearly indicated.

Iron and Phosphorus.—The presence of small quantities of phosphorus in iron and steel produces cold-shortness. A given quantity of this element produces the same degree of

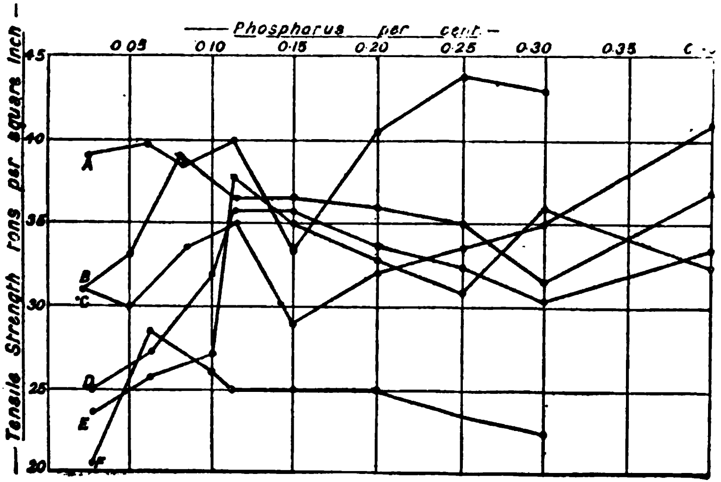

FIG. 3.

cold-shortness as the same amount of sulphur produces red-shortness. The degree of hardening of iron or steel by phosphorus, however, depends upon the proportion of carbon and silicon present. Phosphorus hardens steel to twice the extent as the same quantity of carbon does, and the presence of the element tends to increase the tensile strength of iron and steel, and in the presence of carbon the limit of elasticity is increased. Mr. Howe has studied the influence of phosphorus upon steel in presence of various amounts of carbon from 0·05 to 0·4 per cent. and the diagram, Fig. 3, shows its effect upon the tensile strength of the metal.

PERCENTAGE OF CARBON.

A	0·40 to 0·50
B	0·30 to 0·40
C	0·25 to 0·30
D	0·20 to 0·25
E	0·10 to 0·15
F	0·05 to 0·10

Iron and Tungsten.—Tungsten causes steel to be very hard; steel containing tungsten cannot be tempered except by use of oil. Steel can contain as much as 10 per cent. of tungsten without injuring its forging properties. Tungsten steel is very fine grained, fragile to shock, and has a low limit of elasticity. The metal is very useful for making tools for turning exceptionally hard metal.

Iron and Nickel.—The presence of nickel up to 7 per cent. increases the elastic limit of tensile strength of steel while the extensibility is rapidly diminished. Mr. J. Riley* has investigated the influence of nickel on iron containing from 0·22 to 0·85 per cent. carbon, and 0·23 to 0·85 per cent. manganese, with quantities of nickel varying from 1 to 49·4 per cent. nickel in quantities varying from 8 to 15 per cent. causes such extreme brittleness as not to be properly tested by the usual mechanical means. The following diagram illustrates the influence of various quantities of nickel upon iron:—

* *Journal of Iron and Steel Institute*, No. 1 (1889), p. 45.

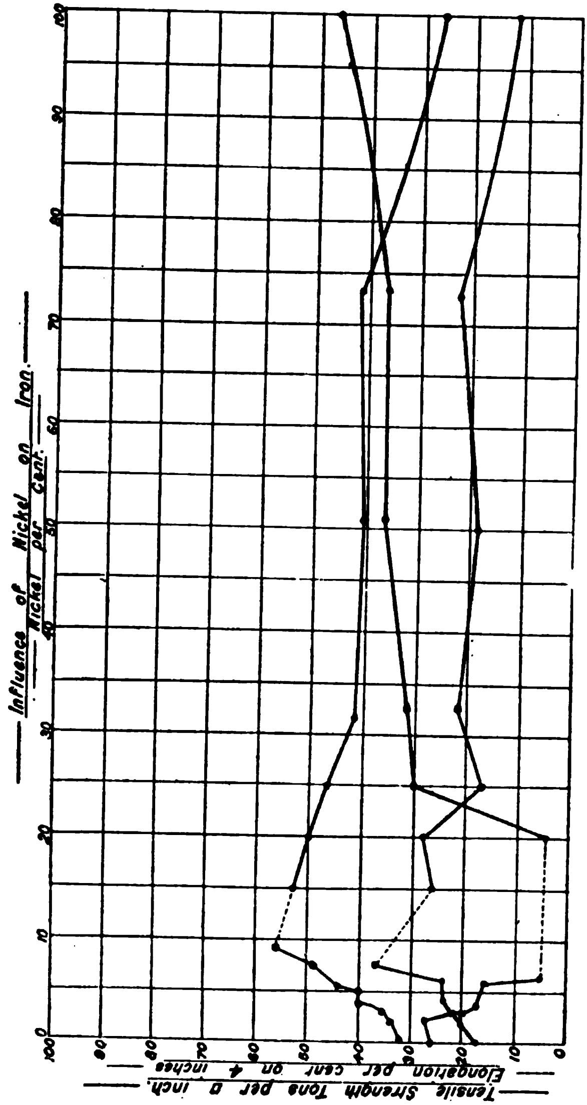
Influence of Nickel on Iron.
Nickel per Cent.
0
10
20
30
40
50
60
70
80
90
100
Tensile Strength Tons per □ inch.
Elongation per cent on 4 inches
100
90
80
70
60
50
40
30
20
10
0

FIG. 4.

Methods for Complete Analysis of Iron and Steel.

Estimation of Silicon, Sulphur, Phosphorus, and Manganese.—Take 8 grms. of sample, brush into 500 cc. beaker, add 70 cc. of 16 E HNO_3, heat gently until red fumes cease to come off, add cautiously 20 cc. of 10 E HCl, boil until all that is soluble is dissolved, evaporate to complete dryness on hot plate, stirring, heat on sand bath for one hour at 200° C. to render silica insoluble, and to destroy organic matter, cool, add 60 cc. of 10 E HCl, and boil until all that is soluble goes into solution. Evaporate to 50 cc., dilute with distilled water to 120 cc., filter off silicon and graphite, remove any adhering to sides of beaker with the aid of a feather, and wash with hot 5 E HCl till free from Fe_2Cl_6 (this is ascertained by allowing some of the washings to drop into a test tube containing a little potassic sulphocyanide solution, when if iron is present a red coloration is produced). Reserve filtrate for the estimation of sulphur, phosphorus, and manganese. Dry the silica, &c., in the water oven, brush into a weighed crucible, ignite filter separately, add residue to main portion, ignite at a bright red heat for a quarter of an hour, cool and weigh the SiO_2, and subtract filter ash from this weight. Remainder $\times \frac{\cdot 4667 \times 100}{8}$ $\doteq$ percentage of silicon present.

This aqua regia method gives results for silicon slightly below the truth. Allen's method, herein described, is the most accurate, although both are frequently used, as is likewise the sulphuric acid method, which gives good results.

Estimation of Sulphur.—The filtrate from the silica is now diluted to 160 cc. in a 200 cc. stoppered graduated test-mixer and thoroughly mixed, and 100 cc. of this (=5 grms. of sample), measured off into a 100 cc. graduated cylinder (the residual 60 cc. is reserved for the determination of phosphorus and manganese) and poured into a 200 cc. beaker. The free acid is neutralized as far as possible with 5 E AmHO (about 10 cc. will be found sufficient, which must be added gradually, stirring the solution thoroughly before each addition) heated to boiling, and 10 cc. of E $BaCl_2$ solution added, stirring. Allow to stand for twelve hours. Decant the supernatant fluid as far as possible from the precipitate, filter the residue through a small "pure" filter paper, wash until free from iron salts with $\frac{E}{10}$ HCl and finally with water, dry in water oven, brush $BaSO_4$ into a weighed porcelain crucible, ignite filter separately, and add the ash. Ignite the crucible and its contents for ten minutes at a dull red heat in an oxidizing flame, cool and weigh the $BaSO_4$, and multiply this weight by $\frac{\cdot 1373 \times 100}{5}$. Result = percentage of sulphur in sample.

A rapid method of estimating sulphur in iron and steel, consists in acting upon 0·2 grm. of the sample with 6 E H_2SO_4 and passing the gas formed through a solution of lead acetate containing 0·2 grm. of the salt per litre, and comparing colour of lead sulphide produced with a sample containing a known quantity of sulphur as estimated by the $BaCl_2$ method.

Estimation of Phosphorus.—Measure off accurately 40 cc. (= 2 grms. of sample) of the original solution as left above (reserving the remaining 20 cc. for the estimation of manganese) into an 80 cc. beaker, add 6 cc. of 16 E HNO_3, evaporate on water bath to dryness, redissolve in 6 cc. of 3 E HNO_3, dilute to 15 cc., gradually add, stirring, 20 cc. of ammonic molybdate reagent (prepared by dissolving 55·5 grms. of molybdenum trioxide (MoO_3) in a mixture of 94 cc. of 20 E AmHO and 150 cc. of water and filtering into 694 cc. of HNO_3 sp.gr. 1·2 = 6·15 E, diluting to 1,000 cc. with 2 E HNO_3, allowing to stand for twelve hours at 50° C. filtering and bottling), allow to stand for three hours at 40° C. with occasional stirring, filter through a small paper and wash until free from iron salts, first with $\frac{E}{10}$ HNO_3 (test the solution to ascertain if precipitation was complete by adding 10 cc. more of molybdate reagent to the filtrate and digesting at 40° C.), and then with water, until free from acid. Spread the filter on a watch glass and dry in water oven, brush off the yellow precipitate of phosphomolybdate of ammonium into a weighed crucible, and dry at 100° C. until weight remains constant. The precipitate as dried at 100° C. contains 1·63 per cent. of phosphorus, so that by multiplying by $\frac{\cdot 0163 \times 100}{2}$ we get percentage of phosphorus in sample.

Estimation of Manganese.—The residual 20 cc. of original solution (= 1 grm. of sample) left from above are poured into a litre flask, diluted to 250 cc., and neutralized with 5 E Am_2CO_3 solution, adding small quantities at a time, and vigorously shaking until a slight permanent precipitate is produced. This is now dissolved by adding two or three drops of 10 E HCl, and then adding 2 cc. in excess; 20 cc. of 5 E ammonic acetate solution are now added, and the solution diluted to 800 cc., and boiled for ten minutes. Filter off quickly the basic acetate of iron $[Fe\overline{A}_3,Fe_2(HO)_6]$ through a large ribbed filter paper into a 1,500 cc. flask, wash quickly half-a-dozen times with boiling distilled water, cool the filtrate to ordinary temperature, add 3 cc. of bromine, shake well until all is dissolved (the object of the bromine here is to convert the manganous acetate in solution into manganic acetate), allow to stand for five minutes and add 20 cc. of 20 E AmHO, boil for ten minutes, filter off the manganic hydrate $[Mn(HO)_2]$ hot, wash with hot water until free from ammoniacal salts, dry in water oven, scrape precipitate into a tared crucible, ignite filter separately, add ash, gradually ignite to full red heat for five minutes, and then at a higher temperature over a blow lamp for five minutes, taking care to avoid reducing gases. Cool and weigh the Mn_3O_4 produced which $\times \cdot 7205 \times 100$ = percentage of manganese present in sample.

Estimation of Graphite and Silicon.—(*Allen's Method.*)—Weigh out 5 grms. of sample into a 200 cc. beaker, add 70 cc. of 5 E HCl, heat until all that is soluble is dissolved, filter into a 500 cc. beaker, wash the precipitate four or five times with $\frac{E}{10}$ HCl, and afterwards three or four times with water. The filtrate is now placed on a water bath to evaporate. Wash the precipitate of silica and graphite into a silver crucible of about 50 cc. capacity, by means of a fine jet of water from a wash bottle, add to it 11 grms. of potassic hydrate, dilute to about 40 cc., put on cover and place in water bath for twenty minutes with occasional stirring. The silica is thus dissolved, the graphite being insoluble, which however always retains a small proportion of silica undissolved. Wash out into a beaker, dilute to about 120 cc. and filter off graphite, adding the filtrate to former on water bath, after previously adding 20 cc. of 10 E HCl. Evaporate to complete dryness and heat on sand bath to 160° C. for one hour, cool, moisten residue with 10 cc. of 10 E HCl, heat on water bath for half an hour, dilute to about 100 cc., boil, filter and wash with $\frac{E}{10}$ HCl till free from iron, and finally with distilled water till free from acid. Dry in water bath, brush into a weighed crucible, heat gradually to full redness for ten minutes, cool in desiccator, and weigh the SiO_2. $\frac{SiO_2 \times {\cdot}4667 \times 100}{5}$ = percentage of silicon.

The graphite on the filter is washed off into a small beaker by means of a jet of water from a wash bottle, and evaporated to dryness on a water bath, after which a mixture of 15 cc. 10 E HCl and 5 cc. 16 E HNO_3 is added, and boiled for ten minutes to dissolve out any iron, &c. Dilute to 60 cc., filter and wash till free from iron. Dry in water oven, brush precipitate into a weighed crucible, and reject filter paper. Heat in an air bath regulated to 160° C. for one hour, cool and weigh. Ignite until all graphite has burned off, cool and reweigh. The residue left here (should not exceed 2 milligrams) is deducted from the graphite, &c., as dried at 160° C., and the difference is taken as graphite, which $\times \frac{100}{5}$ = percentage graphite.

Estimation of Combined Carbon.—There are two methods in use—

(1) *Combustion method*, which involves the separation of the carbon, which is ignited in a current of oxygen, and the resulting CO_2 absorbed and weighed.

(2) *Colorimetric method.* In this method it is assumed that when a steel is dissolved in 6 E HNO_3, the colour of the resulting solution is proportionate to the amount of combined carbon present. A steel in which the combined carbon has been estimated by the combustion method is taken as a standard, and the sample is diluted until the tints are coincident, when the percentage of carbon is easily deduced.

(1) *Combustion Method* (*Total Carbon*).—*Estimation of Combined Carbon and Graphite.*—Weigh out 5 grms. of the sample in a 300 cc. beaker, and pour on 250 cc. of Creath's solution. (This may be prepared in the following manner: Weigh out 170 grms. of pure recrystallized cupric chloride and 107 grms. of pure ammonic chloride, and dissolve in 500 cc. of water, after which run in gradually from a burette $\frac{E}{10}$ NaHO solution, stirring, until a permanent precipitate of cupric hydrate [$Cu(HO)_2$] is produced. Any free acid is thus neutralized. Filter off and dilute to 1,000 cc.) The solution is allowed to act upon the sample for about a quarter of an hour, with frequent stirring, and a gentle heat is applied until all the iron has gone into solution, which can be ascertained by pressing the precipitated copper with a glass rod, and observing if any particles of iron remain undissolved. When this is accomplished, the solution is heated to about 90° C. and kept at this temperature until all the copper has gone into solution, leaving the carbon, &c., suspended in the form of a fine precipitate. It now becomes necessary to filter off the carbon through an asbestos plug, preparatory to its combustion in oxygen. Some pure long-fibre asbestos is ignited at a bright red heat for about half an hour in a porcelain crucible, to destroy any organic matter present, and cooled in a place free from dust. A piece of tubing about an inch in

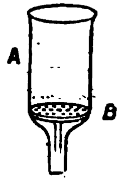

FIG. 5.

diameter is drawn out as in A, Fig. 5, and a disc of perforated zinc, B, fitted into the contraction. Some of the asbestos is now roughly powdered in a clean mortar with a little water, and the whole poured on to the zinc plate, a quantity of asbestos being taken sufficient to make a stratum in the tube about an inch high. Another quantity of asbestos is taken and powdered with water in the mortar very finely, and this is poured on the top of the other until a layer of about a quarter of an inch thick is obtained. This makes a good filter, capable of retaining the most finely divided precipitates. It is now fixed into the cork K of the flask M, Fig. 6, and filled with water, and a gentle stream of water made to pass through the ejector, D, when a partial vacuum is formed in M,

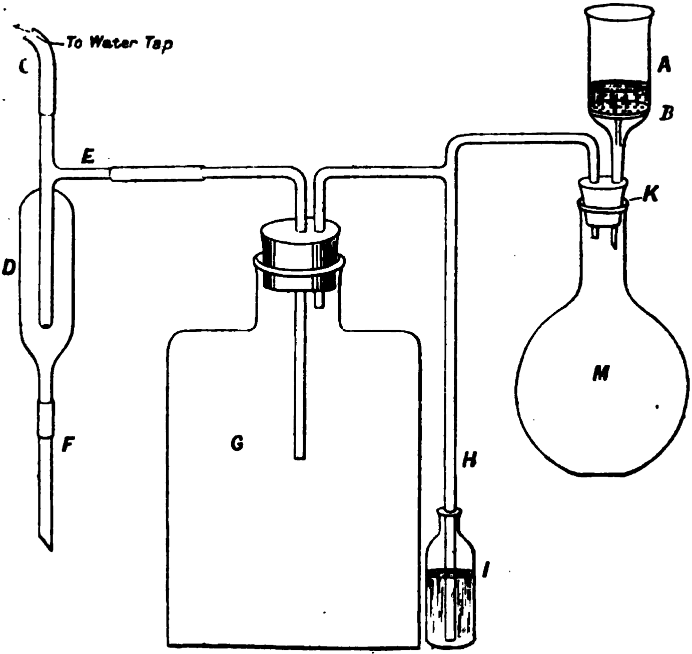

FIG. 5.

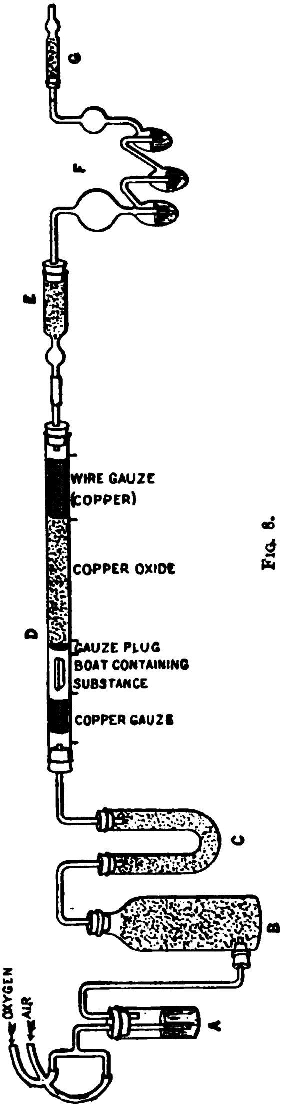

Fig. 8.

and the liquid is soon filtered into M. G is a reservoir, which serves to obtain a steady pull on A, the degree of vacuum being registered by the tube, H, which dips into mercury contained in the bottle, I. The solution containing the precipitated carbon is now filtered through A, with the aid of the ejector (about 3″ of vacuum will usually be found sufficient), and washed with 5 E HCl till free from copper chloride, and the acid afterwards washed out with distilled water, and allowed to drain as far as possible. A is taken

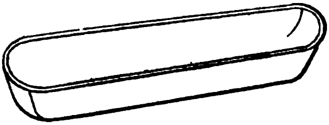

Fig. 7.

out and put into a water oven to dry, B having been previously pushed half-way up the tube; when perfectly dry, the disc and its contents are pushed out of the tube with a glass rod on to a clean watch glass, and the under portion of the asbestos which does not contain any admixed carbon is detached as far as possible by means of a pair of forceps. The remainder containing the whole of the carbon is then placed into a platinum boat (Fig. 7), which is afterwards introduced into the combustion tube, D, in position shown in Fig. 8. As will be seen in the sketch, the tube, besides the boat, contains copper gauze and copper oxide in the proportions seen; the tube should be about 28 in. long and about ½ in. in diameter. The copper oxide should be in a granular form and recently ignited. The cylinder, A, contains 36 E H_2SO_4, and is connected with the tubes X and Y to gas holders containing oxygen and air respectively, and also to the cylinder, B, which is filled with good recently ignited soda lime; C is a U tube containing small pieces of caustic potash. These three tubes serve to absorb any moisture or CO_2 that is present in the oxygen or air that passes through the tube; E is a tube containing $CaCl_2$, which absorbs any moisture that may be given off from the CuO, &c.; F is Giessler's potash bulbs which contain 8 E KHO solution, and G is a small tube containing $CaCl_2$, which serves to absorb any moisture given off from F. When all is ready for combustion, F and G are accurately weighed and attached to E, the tube, D, of course being placed in a combustion furnace. The ends of the tube containing the copper gauze are first gradually heated to redness, a gentle current of oxygen being made to pass through the apparatus. The copper oxide is then gradually heated to its full length to bright redness, and then the part containing the boat is likewise gradually heated to bright redness, the oxygen passing through the potash bulbs about the rate of a bubble a second; after going for about an hour, all the carbon being oxidized, the oxygen is turned off and air turned on at about the same rate; meanwhile the gas is turned out, and after the air has been passing through for about a quarter of an hour, F and G are detached and weighed. Increase in weight $\times \frac{\cdot 27273 \times 100}{5}$ = percentage of total carbon.

Determination of Graphite by combustion.—10 grms. of the sample of steel, or 2 grms. of pig iron are dissolved in 60 cc. of 8 E HCl, and diluted to 120 cc., and the graphite, &c., filtered off in the manner as described for total carbon. When all iron and acid are washed out, some warm E NaHO solution is allowed to percolate through; this is washed out with water and the graphite dried and estimated by the above process. In deducting the graphite thus found from the total carbon, the amount of combined carbon is hereby attained.

The following is an example of an actual determination of graphite and combined carbon in a steel rail by the foregoing process.

TOTAL CARBON.

Five grms. of sample taken.

	Grms.
Potash Bulbs before combustion	37·7658
,, ,, after ,, ...	37·8444
Wt. of CO_2	·0786

$$\frac{\cdot 0786 \times \cdot 27273 \times 100}{5} = \cdot 429 \text{ per cent. total carbon.}$$

GRAPHITE.

Ten grms. of sample taken.

	Grms.
Potash Bulbs before combustion	38·0313
,, ,, after ,, ...	38·0434
Wt. of CO_2	·0121

$$\frac{\cdot 0121 \times \cdot 27273 \times 100}{10} = \cdot 033 \text{ per cent. graphite.}$$

COMBINED CARBON.

$$\cdot 429 - \cdot 033 = \cdot 396 \text{ per cent. combined carbon.}$$

(2) *Estimation of Combined Carbon by Eggertz' Colorimetric Method.* — The principle of this method, as already stated, is, that when steel is dissolved in 6 E HNO_3 a more or less brown colour is produced, which may be taken as being in proportion to the amount of combined carbon present. A standard steel is first selected, the combined carbon in which has been previously determined by combustion. It is important that the amount of carbon present in the standard should be somewhat near to that expected in the sample. It would be wise to keep at least three standards

(1) containing about 1 per cent. carbon for tool steels, &c.; (2) containing ·40 per cent. for rails, tyres, &c.; (3) containing ·20 per cent. for axles and soft steels generally. Suppose we have to determine the combined carbon in a sample of rail steel by this method. Select two test tubes, 5 in. by ½ in., and weigh into them 0·1 grm. of the 0·4 per cent. standard, and 0·1 grm. of the sample respectively, and add to each 2 cc. of 6 E HNO_3. A beaker of water or a copper bath arranged with a perforated false bottom, through which the test tubes can pass, is made to boil, and the test tubes put in and allowed to stay in the boiling water for exactly a quarter of an hour with occasional shaking, after which they are taken out and allowed to cool. The standard is now poured out into a graduated tube (Fig. 9), holding 20 cc. graduated in $\frac{1}{10}$ cc., and diluted to 8 cc., each cc. of which will now equal ·05 per cent. of combined carbon. Pour the sample into a similar tube, and dilute with small quantities of water delivered from a wash bottle, and compare tints after each addition, until the colours are seen to be as near as possible alike. A piece of tissue paper held behind the tubes renders the observation more exact. Suppose that when 6·5 cc. was reached the colours were alike, the amount of carbon present would be 6·5 × ·05 = ·325 per cent.

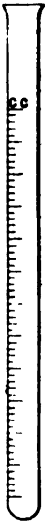

FIG. 9.

Sulphuric Acid Method for the Estimation of Silicon.—The following method will be found to give good results for silicon determinations:—10 grms. of the sample are weighed out into a 400 cc. beaker, dissolved in 120 cc. of 5 E H_2SO_4, evaporated as far as possible on a water bath, and then heated on a sand bath or hot plate until fumes of H_2SO_4 are evolved. The beaker is now cooled, 120 cc. of distilled water poured cautiously on to the residue, the solution boiled up for a short time, the insoluble silica filtered off, washed till free from acid, and determined as directed on page 38.

Rapid Method for Phosphorus Determination.—5 grams of the sample of iron or steel are gradually added to a mixture of 30 cc. of 16 E HNO_3 and 20 cc. of 10 E HCl contained in a 300 cc. beaker; the mixture is then cautiously heated until all that is soluble is dissolved, evaporated to dryness, and the residue heated up to about 200° C. for some time, to destroy organic matter. This is very essential, as the presence of organic matter prevents to some extent the subsequent complete precipitation of the phosphorus. The residue is now dissolved in about 30 cc. of 10 E HCl with the aid of a gentle heat, diluted with distilled water to about 60 cc., and the solution filtered, and received in a 700 cc. Phillips' conical beaker, which is afterwards evaporated to about 20 cc.; 6 cc. of 16 E HNO_3 is now added together with 40 cc. of alkaline ammonic molybdate solution (prepared by dissolving 52 grams of molybdate of ammonia in a mixture of 200 cc. of 20 E AmHo and 800 cc. of water). 20 E AmHo is now cautiously added until the whole of the iron is precipitated as hydrate, and a slight excess of ammonia is present; 16 E HNO_3 is now gradually added, the solution at the same time being vigorously shaken, until all the hydrate of iron is dissolved, and there remains about 5 cc. of 16 E HNO_3 in excess; the conical and its contents are now kept at a heat of about 90° C. for half an hour, after which the phospho-molybdate of ammonia is filtered off and washed free from Fe_2Cl_6, &c., with a one per cent. solution of HNO_3, after which it is dried at 100° C., and weighed.

The weight obtained multiplied by $\cdot 0163 \times 20 = {}^{\circ}/_{\circ}$ of phosphorus. The writer finds that this method, if carefully performed, gives very reliable results. It must be remembered that if there be any arsenic present it will be coprecipitated with the phosphorus precipitate. If it be desired to separate arsenic, this is best done by previous precipitation with sulphuretted hydrogen.

Estimation of Copper.—Weigh 1 grm. of the sample into a 100 cc. beaker, add 12 cc. of 6 E H_2SO_4, heat until all iron has gone into solution, dilute to 50 cc. and boil; now add gradually a solution of 3 E thiosulphate of soda ($Na_2S_2O_3$), as long as a black precipitate continues to form, and on allowing to settle, the supernatant liquid contains suspended sulphur only; (from 1 to 2 cc. of thiosulphate of soda are usually required) the copper is thus precipitated as subsulphide (Cu_2S). Filter off through a small filter and wash until free from iron with distilled water, dry in a water oven, carefully ignite filter and precipitate in a good porcelain crucible, digest with 2 cc. of 10 E HCl + 1 cc. of 16 E HNO_3 until dissolved, evaporate nearly to dryness, add ½ cc. of water, make slightly alkaline with 5 E AmHO, filter off any iron, &c., and receive the filtrate in a carbon tube (Fig. 5), washing the filter a little with 2 E AmHO in small quantities until the blue tint thus produced is the same as that of a standard copper solution contained in a similar tube, and which is prepared by dissolving 0·1 grm. of pure electrotype copper in 1 cc. of 6 E HNO_3 and 2 cc. of 10 E HCl, and diluting to 500 cc. with 2 E AmHO at 15·5° C., 1 cc. thus containing ·0002 grm. of copper. Supposing that the volume of the sample was 5 cc. when the tints were coincident, $5 \times \cdot 0002 \times 100 = 0 \cdot 10$ per cent. of copper present.

Estimation of Iron.—Usually in iron and steel analyses the amount of iron is taken by difference after estimating all the other elements present. It sometimes happens, however, that a direct determination of iron is required, when the following is the best method to adopt. Weigh out ·56 grm. of the sample into a small assay flask of about 80 cc. capacity, add 10 cc. of 10 E HCl, and heat until dissolved; then add in small portions at a time about ½ grm. of potassic chlorate, to oxidize organic matter, &c., and evaporate down to about 4 cc. Transfer to a bulb flask of about 500 cc. capacity, supplied with a rubber cork into which a tube with a Bunsen valve is fitted. Dilute to 200 cc., then add 10 cc. of 2 E Na_2SO_3, and its equivalent of HCl, viz. 2 cc. of 5 E. Attach cork and tube, and boil until free from SO_2, which usually takes about a quarter of an hour or twenty minutes after it starts boiling. Close the valve quickly and put the flask into cold water to cool. The iron is thus reduced to the form of ferrous chloride, as the following reaction shows:—

$$Fe_2Cl_6 + OH_2 + Na_2SO_3 = 2\,FeCl_2 + Na_2SO_4 + 2\,HCl.$$

The next step in the process is to ascertain how much of an oxidizing agent is necessary to convert the $FeCl_2$ into Fe_2Cl_6. In this case bichromate of potash ($K_2Cr_2O_7$) is the most suitable to use.

When the solution is cold, pour it into a beaker of about 500 cc. capacity, and test, to ascertain if perfectly reduced, by bringing a drop of the solution on the end of a glass rod in contact with a drop of E potassic sulphocyanide solution on a porcelain plate, when if a mere trace of Fe_2Cl_6 is present a red coloration will be produced. If no coloration is produced add 10 cc. of 10 E HCl. Fill a 100 cc. burette graduated in 0·1 cc. with *standard* $\frac{E}{10}$ potassic bichromate ($K_2Cr_2O_7$), prepared by dissolving 4·917 grms. of the pure, dry, recrystallized salt in about 500 cc. of pure distilled water, and diluting to 1000 cc. at 15·5° C. (This solution should be standardized by the method here described, taking ·56 grm. of pure pianoforte wire, when 99·7 cc. of the $\frac{E}{10}$ solution should be required, the 0·3 cc. being allowed for unavoidable impurities in the iron). Run the $\frac{E}{10}$ $K_2Cr_2O_7$ into the iron solution at the rate of about $\frac{1}{10}$ cc. per second, with continual stirring, bringing a drop of the solution out from time to time on the end of a glass rod and mixing with a drop of dilute solution (·5 grm. in 500 cc.) of potassic ferricyanide, when a more or less blue coloration is produced as long as there is any ferrous chloride present. When no further blue or green colour is produced with the ferricyanide on allowing to stand for four minutes, the reaction may be considered completed. Note the number of cc.'s required.

The following is the action of the bichromate:—

$$6\,FeCl_2 + 14\,HCl + K_2Cr_2O_7 = 3\,Fe_2Cl_6 + Cr_2Cl_6 + 2\,KCl + 7\,OH_2.$$

Supposing that 98·7 cc. were required, then, since
of the sample was taken and 1 cc. of $\frac{E}{10}$ $K_2Cr_2O_7 =$ ·00
Fe, the amount of iron present is 98·7 per cent.

In addition to the determination of the element
scribed in iron and steel, there are, of course, specia
in which tungsten, chromium, aluminium, or titanium
present. If the character of the iron or steel is unkr
would be safer to make first a qualitative analysis of the
and the result thus obtained would decide what elemen
be determined. The following are methods for the est
of the above-named four elements:—

Estimation of Aluminium.—The following is t
cess adopted by Mr. Stead for estimating small quan
aluminium in iron and steel:—Weigh out 11 grms. of the
in a 600 cc. beaker, add 44 cc. of 10 E HCl, heat u
solved, and evaporate to dryness. Redissolve residue i
of E HCl, filter off into a 500 cc. beaker, and wash the
till free from iron. Dilute the filtrate to 150 cc., add
E sodic phosphate solution, and gradually add 5 E
until the free acid is neutralized and a permanent pre
is produced. Now add 10 E HCl drop by drop until t
tion has cleared up, heat to boiling and add 50 cc. of
rated solution of sodic thiosulphate, and continue bo
one hour; filter and wash. Dissolve the precipitate
filter with 40 cc. of hot 5 E HCl, collecting the filtr
platinum dish, wash the filter and evaporate to dryne
2 grms. of sodic hydrate and 1 cc. of water; allow to d
evaporate to dryness, and fuse for about ten minutes
Bunsen. Cool and digest with 50 cc. of water, and
to exactly 110 cc.; filter off the insoluble oxides ar
sure off 100 cc. of the filtrate (= 10 grms. original s
neutralize with 10 E HCl, add 3 cc. of E sodic phosph
30 cc. of the saturated thiosulphate solution, and boi
add 2 cc. of 5 E ammonic acetate, and boil five minutes
and filter; wash the precipitate with hot water, dry

and weigh the $AlPO_4$. $AlPO_4 \times \cdot 2236 \times 10 =$ percentage of aluminium.

Estimation of Chromium.—(*Arnold's Method.*)—Weigh out 2 grms. of the sample into a 100 cc. beaker, add 20 cc. of 10 E HCl, and heat until dissolved; evaporate over the water bath to complete dryness; detach as much as possible of the crisp residue with the aid of a glass rod, and brush on to a clean watch glass; any chloride still clinging to the beaker is dissolved off with a few cc. of 5 E HCl, and poured into a deep platinum crucible and evaporated to complete dryness. The main portion of the chlorides on the watch glass is now brushed into the crucible and the whole finely powdered with a glass rod; 5 grms. of fusion mixture (1 part Na_2CO_3 and 1 part KNO_3) are now introduced and thoroughly mixed with the chlorides. The mixture is now fused for about fifteen minutes over a blow-lamp. The iron is thus converted into insoluble oxide, and the chromium, silicon, and manganese into alkaline chromate, silicate, and manganate respectively. Allow to cool, put the crucible and contents into a beaker containing about 80 cc. of boiling water, and digest until all that is soluble is dissolved; take out the crucible with the aid of a glass rod, and wash the liquid adhering to it into the beaker with hot water. Now add 1 cc. of alcohol to the solution to decompose the manganate, and stir well and allow the oxides of iron and manganese to thoroughly settle. Decant the clear liquid through a double filter (without disturbing the precipitate) into a beaker; add 30 cc. of boiling water to the residue, stir, allow to settle, drain off the clear liquid through the filter as before, wash the filter twice with hot water, and treat the residue with a second 30 cc. of hot water and filter as before; this may be considered sufficient washing for the oxides. The solution, which now contains chromate and silicate, is acidified with 20 cc. of 10 E HCl and boiled until all CO_2 and nitrous fumes have come off. Now add gradually 5 E AmHO until slightly alkaline, heat nearly to boiling, and filter off the resulting precipitate through an ashless filter and wash. Dissolve the precipitate off the paper with 10 cc. of hot 5 E HCl, wash t into the solution and evaporate to complete dryness on bath to render the silica insoluble; dissolve residue in 1 E HCl, filter off silica, collecting filtrate into a porcelai neutralize the solution with 20 E AmHO, adding about excess and heat to incipient boiling, filter off the hydrate $Cr_2(HO_6)$ through an ashless filter, wash with water till free from soluble matter, dry in water oven, an carefully in weighed crucible for ten minutes at a good r Cool, and weigh the resulting Cr_2O_3.

$$\frac{Cr_2O_3 \times \cdot 6862 \times 100}{2} = \text{per cent. chromium.}$$

Estimation of Titanium.—(*Riley's Method*).—W 10 grms. of the sample into a 500 cc. beaker, pour 60 cc. of 16 E HNO_3, and heat on a hot plate until i on the iron has nearly ceased; now add cautiously 10 E HCl, boil until all that is soluble is dissolved rate to dryness, heat the residue on a sand bath t 130° C. for an hour, cool, add 50 cc. of 10 E HCl, until all that is soluble is dissolved. The solutio should now be about 40 cc., is diluted to 80 cc., graphite and silica, with some titanic acid, are filtered washed with hot $\frac{E}{10}$ HCl till free from iron. The filtr served, the residue is dried and ignited in a platinum then fused with about 16 times its weight of bisul potash, and then cooled and digested with hot water that is soluble is dissolved; it is now filtered, and th added to the reserved filtrate containing the iron, & the mixed filtrates into an 800 cc. flask, provided wit fitted with a Bunsen valve, dilute to 300 cc., add 1 2 E Na_2SO_3 and 20 cc. of 10 E HCl, and boil until solution is reduced and all sulphurous odour has Cool, add 1 cc. of E HNO_3, nearly neutralize with add 80 cc. of 5 E ammonic acetate, boil for a few and filter off the precipitate quickly, which contains titanic acid, oxide of iron and phosphate of iro with $\frac{E}{10}$ acetic acid, dry, ignite in platinum crucible, and fuse with 16 times its weight of bisulphate of potash; digest in cold water, and filter off phosphate of iron, &c. Dilute the filtrate to 500 cc., add 1 cc. of 16 E HNO_3, and boil for about six hours, keeping up the original volume with additions of boiling water. The titanic acid is now allowed to settle over night, the clear liquid decanted, the residue filtered, washed, dried, and ignited in a platinum crucible, and then cooled and weighed. The weight of TiO_2 thus produced × ·6098 × 10 = percentage of titanium.

Estimation of Tungsten.—Weigh out 5 grms. of the sample, dissolve in 120 cc. of 6 E HNO_3, evaporate to dryness on sand bath, cool, add 40 cc. of 10 E HCl, and boil until all that is soluble is dissolved; add 40 cc. of water and again boil, filter off the precipitate, which may consist of oxide of tungsten (WO_3), graphite, Fe_2O_3, TiO_2, &c., wash with hot water, dry and ignite in a platinum crucible; add 10 cc. of hydrofluoric acid (HF) and 2 cc. of 36 E H_2SO_4, evaporate in a hood, and repeat the evaporation with HF and H_2SO_4 until all SiO_2 has escaped as SiF_4; add to the residue four times its weight of Na_2CO_3, fuse over a Bunsen for ten minutes, cool, and treat the fused mass with water until all that is soluble is dissolved. Filter off insoluble oxides, &c., and wash with $\frac{E}{5}$ Na_2CO_3. The solution now contains all the tungsten as soluble tungstate of soda. Dilute to 100 cc., neutralize with 16 E HNO_3, boil off CO_2, and add two drops of 16 E HNO_3 in excess; add 50 cc. of $\frac{E}{5}$ mercurous nitrate, neutralize the free acid by adding sufficient recently precipitated oxide of mercury, and stir well. Allow to settle, filter off the mercurous tungstate with the slight excess of oxide of mercury, and wash with hot water till free from soda salts; dry in water oven, scrape into platinum crucible, ignite filter separately, and add ash. Now heat the crucible under a hood gradually to a bright red heat, until fumes

leaves the tungsten in the form of oxide (WO_3). C
weigh.

$$\frac{WO_3 \times \cdot 7931 \times 100}{5} = \text{percentage tungsten.}$$

ANALYTICAL DATA OF AN ACTUAL ANALYSIS, BY METHODS, OF BAD SAMPLE OF BESSEMER STE

ESTIMATION OF COMBINED CARBON.

(*Eggertz' method.*)

0·1 grm. of sample taken.

The total volume of solution of sample, when the
equal to the standard solution (half strength) was 8·6
Now each cc. of the *dilute standard* solution contain
grm. of carbon, therefore,

$$\frac{8{\cdot}6 \times \cdot 00005 \times 100}{0{\cdot}1} = 0{\cdot}43 \text{ per cent. combined carbo}$$

ESTIMATION OF SILICON.

(*Sulphuric acid method.*)

10 grms. taken.

Porcelain Crucible marked 6 . . . . .
Do. + SiO_2 + F. Ash . . . . . .
" " " 2nd ignition . . . .

F.A. . . .

SiO_2 = . . .
SiO_2 into Si . .

·0

$$\frac{\cdot 0304 \times 100}{10} = 0{\cdot}304 \text{ per cent. of silicon.}$$

ESTIMATION OF SULPHUR.

5 grms. taken.

	grms.
Porcelain Crucible marked 7	7·3390
Do. + $BaSO_4$ + F.A. + *$BaSO_4$ from reagents* used .	7·3633
„ „ „ „ 2nd ignition	7·3631
$BaSO_4$ + F.A. + Re	·0241
F.A. + Re	·0019
$BaSO_4$	·0222
$BaSO_4$ into S	·1373

666
1554
666
222

·00304806

$$\frac{\cdot003048 \times 100}{5} = 0\cdot061 \text{ per cent. of sulphur.}$$

ESTIMATION OF PHOSPHORUS.

2 grms. taken.

	grms.
Porcelain Crucible marked *x*	3·7960
Do. + "ppt" dried at 100° C	3·8242
„ „ „ 2nd drying . . .	3·8242
	·0282
"ppt" into P . . .	·0163

846
1692
282

·00045966

$$\frac{\cdot0004596 \times 100}{2} = 0\cdot023 \text{ per cent. of phosphorus.}$$

ESTI

Porcelain Crucible ma
Do. + Mn_3O_4 + F. A
" " "

·009438 × 10

E

Volume of blue sol
standard = 3·75 cc.
1 cc. of *Sta*
therefore,
3·75 × ·0002

ANALYSES O

ANALYSIS OF A SAMPL

Iron .
Nickel
Cobalt
Copper
Tin .
Manganes
Phosphor
Sulphur
Chlorine
Carbon
Silica

ANALYSES OF PIG IRONS.

	Northampton. Hot Blast.	Hematite. Bessemer Pig No. 1.	West Hallum. No. 3 Foundry.	Kirkless Hall. No. 3.	Silicious Pig Iron.
Graphite . .	1·150	3·050	2·650	3·200	1·80
C. Carbon . .	·554	·710	—	—	0·20
Silicon . .	1·900	2·012	1·350	1·275	9·17
Sulphur . .	0·414	0·010	0·055	0·081	0·01
Phosphorus .	1·807	0·052	0·650	1·750	0·06
Manganese .	0·395	0·040	0·415	2·215	2·86
Iron (by difference)	93·780	94·126	94·880	91·479	85·90
	100·000	100·000	100·000	100·000	100·00

ANALYSES OF VARIOUS CAST IRONS.

	Gurlt.			Bodemann.		Abel.	
	Grey Coal.	Mottled Hot Blast.	White Gartsherrie.	Grey Hot Blast.	Mottled Cold Blast.	Grey. French Charcoal.	White Silesian.
Density .	7·21	7·21	7·41	7·166	7·43	7·000	7·531
Carbon combined .	1·021	1·793	2·457	1·44	2·78	—	4·94
Graphite .	2·641	1·110	0·871	2·71	1·99	3·40	—
Silicon .	3·061	2·165	1·124	3·21	0·71	0·80	0·75
Sulphur .	1·139	1·480	2·516	trace	trace	0·05	trace
Phosphorus	0·928	1·171	0·913	1·22	1·23	0·45	0·12
Iron . .	90·236	89·314	89·863	91·42	93·29	95·18	88·57
Manganese .	·834	1·596	2·715	trace	trace	—	5·38
Copper .	—	—	—	—	—	—	0·24
	99·860	98·629	100·459	100·00	100·00	99·88	100·00

ANALYSIS OF A BAD SAMPLE OF BESSEMER STEEL RAIL BROKEN BY WEIGHT OF LOCOMOTIVE.

Combined Carbon . . .	0·275
Silicon	0·235
Sulphur	0·037
Phosphorus	0·065
Manganese	0·750
Iron (by difference) . . .	98·638
	100·000

The high percentage of silicon in this rail was no doubt the cause of breaking.

Analysis of a Bessemer Steel Rail that was Broken during Transference from Rail-bank to Waggon.

Combined Carbon	0·350
Silicon	trace
Sulphur	0·080
Phosphorus	0·230
Manganese	0·150
Iron (by difference)	99·190
	100·000

The high percentage of phosphorus, making the steel "cold short," was no doubt the cause of breakage. Often chemical analysis does not show any reason why a rail should break. In these cases it is either due to a flaw, or to a want of physical uniformity resulting often from careless manufacture.

The author considers that an ideal rail and tyre should have the following compositions, provided that a thorough homogeneous metal is insured:—

	Rail.	Tyre.
Combined Carbon	0·350 to 0·400	0·400 to 0·500
Silicon	0·056 ,, 0·070	0·080 ,, 0·100
Sulphur	0·040 ,, 0·060	0·040 ,, 0·060
Phosphorus	0·040 ,, 0·060	0·040 ,, 0·060
Manganese	0·750 ,, 1·000	0·400 ,, 0·600

Analysis of a Krupp Tyre that has done Good Service.

Combined Carbon	0·500
Silicon	0·275
Sulphur	0·035
Phosphorus	0·060
Manganese	0·475
Iron (by difference)	98·655
	100·000

It will be observed that this tyre contains a high percentage of silicon, and although it wore well it would be unsafe to generally recommend such steel for the purpose, especially if manufactured by the Bessemer process. With 0·5 per cent. of carbon in a tyre the amount of silicon should not exceed 0·1 per cent.

The following results show that the elements are not evenly distributed in an ingot (Snelus) :—

	Top of Ingot.	Bottom of Ingot.
Combined Carbon	0·760	0·350
Silicon	trace	trace
Sulphur	0·187	0·044
Phosphorus	0·191	0·044
Manganese	0·558	0·514

Six samples were taken out of the top of an ingot and six out of the bottom from positions sketched in section (Fig. 10), when the following results were obtained :—

	From Section at Top.			From Section at Bottom.		
	Carbon.	Sulphur.	Phosphorus.	Carbon.	Sulphur.	Phosphorus.
1	·44	·032	·044	·44	·048	·060
2	·54	·048	·066	·42	·056	·062
3	·57	·080	·086	·41	·048	·054
4	·61	·096	·097	·40	·048	·054
5	·68	·120	·111	·38	·048	·058
6	·77	·187	·142	·37	·044	·052

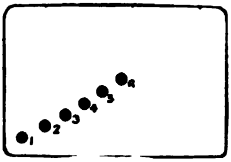

Fig. 10.

Analysis of Steel used for Dies in Royal Mint.

	(1)	(2)	(3)	(4)
Carbon (combined)	0·82	1·07	0·79	1·19
Manganese	0·10	0·12	0·24	0·45
Silicon	0·05	0·06	0·18	0·29
Sulphur	trace	trace	0·01	trace
Phosphorus	—	,,	0·01	,,

ANALYSIS OF SWEDISH GUN-BARREL STEEL.

Carbon	0·450
Silicon	0·450
Manganese	0·400
Sulphur	0·040
Phosphorus	0·085
Iron (by difference)	98·575
	100·000

ANALYSIS OF TWO SAMPLES OF KRUPP-GUN STEEL OF APPROVED QUALITY.

	(1)	(2)
Combined Carbon	·42	·69
Graphite	·04	·02
Silicon	·11	·06
Manganese	·13	·15
Phosphorus	nil	trace
Sulphur	—	—
Copper	·30	·26

SPECIFICATION OF QUALITY OF STEEL SUPPLIED TO THE COCKERHILL WORKS, SERAING, FOR THE PURPOSE OF SHIPBUILDING.

Carbon	·08 to ·15
Silicon	trace „ ·02
Sulphur	·03 „ ·05
Phosphorus	·03 „ ·05
Manganese	·30 „ ·60

TABLE SHOWING THE WEAR AND COMPOSITION OF STEEL RAILS.

Wear.	Time in use.	Carbon.	Silicon.	Sulphur.	Phosphorus.	Manganese.
*$3\frac{1}{16}''$	4 years	·50	·185	·036	·053	1·138
$3\frac{1}{16}''$	„	·41	·035	·084	·071	·980
$\frac{3}{8}''$	„	·27	·016	·084	·047	·331
$5\frac{1}{16}''$	„	·37	·055	·093	·060	·540
$7\frac{1}{16}''$	12 „	·54	·026	·027	·060	1·166
$9\frac{1}{16}''$	10 „	·40	·046	·037	·029	·958
$5\frac{1}{16}''$	8 „	·45	·053	·127	·063	·677

* Broke.

Application of the Microscope to the Study of the Structure of Iron and Steel.—The microscopical examination of samples of iron and steel, as a means of ascertaining the nature of structure, &c., as bearing upon the physical properties of the metal, has of late received considerable attention from chemists and engineers, and in the result will, no doubt, eventually have a far-reaching influence in the economic conduct of metallurgical operations.

I am indebted to Mr. J. E. Stead, F.I.C., F.C.S., of Middlesbrough, for a description of his admirable system of practical metallography, and the beautiful photographic illustrations I am able to reproduce here.*

The operations involved consist in (1) Preliminary preparations of specimens; (2) Methods of polishing; (3) Etching the specimens; (4) Mounting the specimens; (5) Photographing the developed structures.

Preliminary Preparation of Specimens.

When time is no object and labour of little account, it is advisable to obtain all the metal sections in the form of little plates about $\frac{3}{8}$ to $\frac{1}{2}$ in. in diameter, and $\frac{1}{10}$th of an inch thick. This can easily be managed if the metals are soft enough to be sawn, filed, or turned into shape, but it is very difficult, unless a diamond cutter is available, to obtain harder metals of such dimensions. It is, however, only necessary to have one plane surface, and the irregularity of the other portion is immaterial. In preparing pieces of white pig iron and other hard brittle metals or alloys, it is only necessary to take a chip or fragment not more than $\frac{1}{8}$ in. thick, with one smooth surface. This face is ground on an emery wheel till it is flat, then finished as will be described.

It saves much time, and an emery wheel can be dispensed with, if the surface to be polished does not exceed $\frac{1}{4}$ in. in diameter; emery paper will be capable of grinding to flatness such a small area.

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* For the loan of the blocks of the illustrations of photographs Nos. I. to VII. I have to thank the Council of the Cleveland Institution of Engineers.

When sections of wires are to be examined, take a small piece of hard steel ¼ in. round by $\frac{1}{10}$ in. in thickness, and drill a hole through the centre just large enough for the wire to fit it (Fig. 11).

The wire is soldered at the back to its support, care being taken to keep the face, with the wire protruding, cool, by placing it on a metal block when soldering. The wire can be cut off close to the back of the support. The face of the piece is then ground and polished. The end of the wire remains quite flat after polishing.

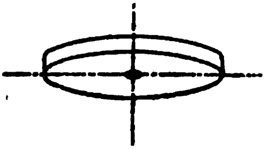


FIG. 11.

In supporting sections of thin plates, pieces of steel about ½ in. square and ¼ in. deep are cut two-thirds through with a hack saw (Fig. 12).

The thin plate is inserted into the cut, and the sides of the supporting steel are pressed against the specimen by pressure in a vice or by a sharp blow from a hammer. If necessary, three or four pieces can be mounted in the same block. When thus secured, the specimens are ready for polishing. In examining the worn surfaces of rails or tyres, which are often covered with microscopic cracks, it is clear that such surfaces must not be ground down to obtain a perfect polish, for by

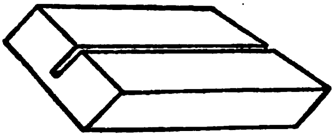


FIG. 12.

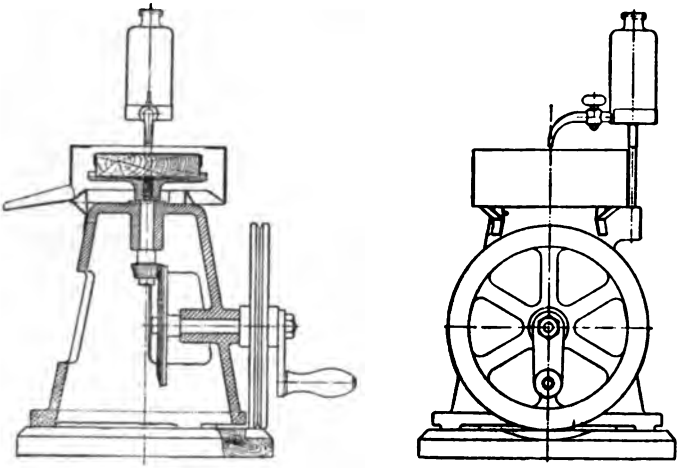
doing so the area in which the cracks are situated would be removed. It is therefore advisable to polish with the finest emery paper, No. 0000, and to finish on rouged leather. This treatment will leave a very rough surface, pitted with rust marks, but quite clean enough for the purpose. Probably the cracks will be visible under a strong lens without any previous treatment with acid, and if not by a lens, by a low power objective under the microscope. If nothing can be detected after polishing alone, the specimen must then be allowed to remain for thirty seconds in a 1 per cent. solution of strong nitric acid in water, after which it is washed in distilled water, then soaked in lime water, and again washed with water, and finally polished on rouged chamois leather. It is then examined, and the cracks will then probably be seen: if not, immerse again in acid and proceed as before. If no cracks appear, soak in a 5 per cent. solution of nitric acid (5 cc. HNO_3, sp. gr. 1·42, and 95 cc. of water) for one minute, wash and treat as before. If now no cracks are visible they may be considered to be absent.

In the preparation of a vertical section, after having cut out a horizontal slice about ¼ inch in thickness, and ¼ inch in width, and ¾ inch in length; cut it vertically into two halves, so as to make two pieces each ¾ in. long, 3/32 in. wide, and ¼ in. thick. Rub the two head surfaces on the finest emery paper to remove excrescences produced by the saw; then place them against each other and screw them in that position in a vice, taking care to have the two pieces exactly parallel. Now smoothly file the upper surface projecting from the vice, and dust with resin powder, and with the soldering-iron drop a piece of very fusible solder on to the filed surface, and apply the soldering-iron until it unites with the steel. This should be done so rapidly as not to cause the steel to be materially heated. Now turn the soldered side down and secure in vice, file smooth, remove from the vice, and grind on emery, and finish on cloth and rouged parchment or leather. You will now have a specimen quite flat across the centre part where the two head surfaces rest one against the other, and you will have two vertical sections, the fine line in the centre, of course, being the division between them.

The object of placing the two surfaces together in this way is to avoid the slight rounding or curving at the parts where it is most important they should be perfectly flat. If the precaution is not taken when polishing on soft beds, the edges get worn down, and are then unsuitable for examination, especially under high powers.

Mechanical Device for Polishing.

Figs. 13 and 14 show the machine designed by Mr. Stead



FIGS. 13 AND 14.

for polishing specimens which may be procured from Messrs. Carling and Son, Middlesbrough. It consists of—

(1) A horizontal revolving wheel driven by hand at the right of the machine.

(2) Conical blocks with rings for securing in position the emery papers, &c. There are several of these blocks for carrying the different polishers. They are simply

dropped into the recessed wheel and are carried round by friction.

(3) A copper can for holding either water or a solution of ammonia nitrate, and a tap for allowing the solution to drop on to the centre of the polishing blocks.

(4) A shield for catching the fluid which is thrown oft the wheel when it is at work.

(5) Cases for the blocks, to prevent dust falling upon them when not in use.

In describing the *modus operandi* of Mr. Stead's system, I cannot do better than reproduce the record of the practical demonstration given by him.* Taking for his trial a ⅜ bar of steel, containing about 0·40 per cent. carbon, he proceeded as follows:—

"1.—The bar I place in a vice and file one end quite flat with a smooth file.

"2.—It is now fixed into a second vice attached to a hack saw and a section is cut an eighth of an inch in thickness.

"†3.—A No. 0 emery paper block is placed on the machine, and with the first finger of the left hand the specimen is pressed on the block whilst the wheel is being revolved with the right hand. The specimen is kept in one position till all the file marks have disappeared, and the emery scratches are all in one direction. The specimen is now turned 90°, and the pressure having been reduced the wheel is again made to revolve till again the scratches are in one direction, but at right angles to those first made.

"4.—A second block carrying finer emery paper, No. 00, is now placed on the wheel, and the same treatment given to the section as just described under No. 3.

"5.—A still finer block is now used, No. 000, and the same treatment repeated.

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* Before the Cleveland Institution of Engineers on February 26th, 1900, in the Hall of the Cleveland Literary and Philosophical Society at Middlesbrough.

† The emery papers are those used by steel engravers for polishing their steel plates. They are made in France. They can be obtained through any good ironmonger.

"6.—The next block is covered with smooth black cloth, and upon its surface is rubbed about one grain of diamontine powder (used by jewellers for polishing steel). Water is allowed to drop on the block from the copper bottle, and the wheel made to revolve. The specimen is pressed on the block and made to traverse across it so as to produce even polishing.

"It is examined from time to time in a good light till all the fine emery scratches are removed. The polish at this stage is sufficiently good for most practical purposes, but if a photograph is required it must be passed over a finer block.

"7.—The final block, suitable for this steel section, consists of chamois leather and fine gold rouge. The rouge is well rubbed in, and then the wheel is caused to revolve rapidly, whilst the surface is pressed with the flat edge of a thin strip of steel. This loosens the coarser particles of rouge, and they are thrown off the block, and none but the finest parts remain. The specimen is now placed on this block, and very gentle pressure applied and the wheel revolved till, on examination, the surface appears to have slightly lost its brilliancy. If the polishing is carried too far, the fine surface is destroyed, and it becomes very uneven and dull.

"The polishing has been stopped at the right moment, and it is ready for etching.

"8.—For finer structures the dry block with rouge is not good enough, and No. 7 is replaced either by parchment or by fine kid leather, the latter being recommended by Professor Arnold.

"In either case the block is first soaked in water, and then the rouge is well rubbed into the surface; the rubbing is continued under a stream of running water, which effectually removes all but the finest particles of the rouge (this is Osmond's excellent plan). The block prepared in this way is placed on the wheel, and the section of metal pressed upon the surface. Water in just sufficient quantity is allowed to drop on the block so as to keep it quite wet, but not sufficient to cause it to flow off.

"It takes longer to polish on wet blocks than on dry, but the results are much superior.

"9.—In making the 'polish attack' of Osmond, parchment is used on the block, but instead of rouge and water, sulphate of lime (prepared by adding a boiling solution of sulphate of ammonia to a hot solution of calcium chloride and thoroughly washing the precipitate) and liquorice root solution are used.

"Treated in this way many specimens of steels and other metals have their structures developed most perfectly without any further treatment.

"Cementite, Pearlyte, Martensite, and Troostite, and what M. Osmond calls Sorbite, are clearly developed by this method.

"MM. Osmond and Carband in the January (1900) issue of the American quarterly journal, the *Metallographist*, give new re-agents for the micrographic study of Carburized irons. They say:—

"A dilute solution of nitrate of ammonium (two parts in weight of the crystallized salt to 100 parts of water) yields good results. A piece of parchment spread tightly over a smooth board is soaked with this solution, and the polished surface of the specimen is rubbed upon it till sufficiently etched. It is not necessary to add any sulphate of calcium. When the parchment becomes dry a little water is poured over it.

"The results are exactly those produced by the extract of liquorice.

"The various constituents are identified as follows:—

"1. Pearlyte by the unequal depth of etching of its two components, and Sorbite by its colouration varying from light yellow to dark brown.

"2. Troostite by its yellow, brown, or blue bands merging into each other.

"3. Martensite by its characteristic needles, which are the better defined the less carbon in the metal.

"4. Ferrite by its division into grains and the heterogeneous appearance of these grains.

"Austenite, and especially Cementite, remain unacted upon.

"It must not be supposed, however, that this combined etching and polishing method by means of nitrate of ammonium is free from all difficulty, nor must it be expected to succeed at the first trial. The re-agent has a tendency to form a film of oxide, which must be removed by rubbing as soon as it is formed, and success depends upon a complex set of conditions (concentration of the solution, pressure, speed, quality of the parchment, etc.), several of which belong to the personal equation, and could not be stated accurately. Every one must serve an apprenticeship, but once the routine of the method acquired, Martensite may be resolved in 400 frictions, back and forth, that is, in less than three minutes of effective work.

"A satisfactory means of distinguishing Austenite from Martensite was still to be found. It can be accomplished by immersing the metal in a solution of chloride of ammonium, a 10 per cent. solution for instance, while it is connected with the positive pole of a bichromate cell, the negative pole consisting of a piece of platinum foil, or of lead, or iron foil. The specimen is examined every ten seconds until the etching is sufficiently deep. A very dilute solution of hydrochloric acid may also be used, with or without the assistance of an electrical current, but the use of a battery is always desirable as it increases the regularity of the chemical action. In both cases Martensite is first coloured, successively yellow, brown, and black."

"In concluding this section, you have observed that the polishing and etching of this sample has not taken more than fifteen minutes, and I can assure you that when the operator is not obliged to talk as well as work, the time need not exceed ten minutes. As a matter of fact, the average time taken in polishing the thousands of sections I have prepared has not exceeded ten minutes. A novice, at first, will probably take half-an-hour or more, but in time, when experience teaches how to graduate the pressure in polishing, the shorter period will suffice. The cutting of suitable sections from a large mass of steel may take a whole day; such preliminary work must be done in a machine shop.

"The following facts should, however, be noted :—

"(*a*) The softer the metals the lighter must be the pressure, and they must never be polished on dry blocks. Lead is the most difficult of all metals to polish, and it is only by the slightest friction long continued that any good polish can be obtained.

"(*b*) Hardened steels and white irons require much greater pressure, and are easily polished.

Etching the Specimens of Iron and Steel.

"1.—The 'polish attack' of Osmond has already been described. It takes the first place, and gives the most perfect results in very many metals and alloys.

"2.—The second method of etching steel is by a tincture of iodine of the following strength :—

Iodine	1·25 grams.
Iodide of Potassium	1·25 "
Water	1·25 "

'Alcohol to make up to 100 cc.

"This is the re-agent used by Osmond. A second weaker tincture ½ the above strength is very useful.

"The polished specimen is placed on a plate, and one drop per square centimetre is allowed to cover it. It is left there until the iodine colour is gone. The specimen is washed in water, then in alcohol, and finally dried in a blast of hot air. It is mounted and examined. The iodine treatment is repeated if the first application has not sufficiently developed the structure.

"Some metallographers advise the wiping of the specimen with a clean linen cloth instead of drying, but this occasionally removes coloured deposits which are sometimes a most important indication. After drying by hot air and examining under the microscope, the specimen may be afterwards wetted, and then dried with the cloth and re-examined.

"3.—Etching with dilute Nitric Acid is often recommended, but it is not so safe for steels as tincture of iodine, but in the absence of much phosphorus and arsenic it gives good results.

"The specimen is immersed in a vessel containing a 0·10 per cent. Nitric Acid (1·42 sp. gr.) in water, and is retained there until the structure is developed sufficiently clear. The progress of the etching should be watched through a strong magnifying glass. After removing from the acid it should be well washed by running water, immersed in lime water, re-washed in water, then in alcohol, and finally dried with hot air. Fletcher's hot blast blow-pipe answers well for this purpose.

"4.—Osmond recommends a 20 per cent. solution of nitric acid for rapidly developing the crystalline or granular structure of steels.

"The specimen is immersed in the acid from one to ten seconds, and for even longer periods, then removed, washed as before, and examined. This I have found to be very useful when deep etching is required.

"5.—Prof. Behrens and Prof. Martens have used, with great success, the simple process of heating the steel and other specimens till they take oxidation tints. This system may be described as a '*heat tinting*' one, as it can scarcely be called etching.

"I have latterly used the method with most satisfactory results, and have found that it is the only method by which phosphides and carbides of iron can be distinguished from one another when associated in iron and steels.

"In preparing the specimen for heat tinting, the bright surface should be well rubbed with a clean piece of linen or chamois leather. Care must be taken to avoid any condensation of water on the surface; this is best avoided by warming the specimen to 90° or 100° C. before giving it a final vigorous rubbing with the linen or chamois leather, and before it cools it should at once be placed on the iron plate. A sheet of iron 6 in. square placed over a Bunsen burner is all that is required for heat tinting. The section is placed in the centre, and the polished surface watched till the proper tint is obtained. Practical experience will soon enable one to find out the best tint for any particular metal or alloy. It is best to heat gradually and examine periodically under the microscope, and stop when the structure appears to be most perfectly coloured. After each heating the section should be placed in a dish containing mercury, so as to cool it rapidly and check further oxidation. If it is desired to photograph the heat-tinted object, it is most important in many cases to stop the heating when the object assumes a pale yellow tint, for although the contrasts to the eye are not so great, they are quite great enough for the sensitive photographic plate.

Mounting the Specimens.

"When the specimens are quite flat, they may be mounted with Canada balsam or wax, upon the glass slips. As it is rarely (unless the specimens have been prepared in the lathe) that the two sides are parallel, it is necessary to provide some means of mounting so as to have the polished surface perfectly horizontal with the glass slide on which it is mounted.

"After trying many plans I find that the simplest method consists in placing the polished face of the section on a piece of plate glass, and placing over it a short brass cylinder accurately turned of such depth that the back of the specimen does not stand above the cylinder. A piece of plastic wax, used in kindergarten schools for making models, is stuck upon the centre of a glass microscope slide, and this is then pressed upon the section till the glass slide comes in contact with the brass ring. The specimen adheres to the wax, and the mounting is complete. It may be made more secure by dressing up the wax against the section and glass plate with a hot wire.

"After examination, if it is desired to preserve the section, I prefer to cover it with a solution of paraffin wax dissolved in benzole. The benzole slowly evaporates, leaving a thin protective coating of wax. It will prevent the formation of rust, and when it is required for re-examination, the wax is dissolved off by wiping it with a clean linen rag moistened with benzole. It is finally gently rubbed with a dry linen rag.

"The brass cylinder rings can be made by any fitter by cutting up a brass tube 1" internal diameter in a lathe, into lengths of ″0·1, ″0·15, ″0·20, ″0·25, ″0·35, ″0·40, ″0·45, ″0·50, ″0·60. They must be accurately faced, after being cut. Messrs. Carling & Son have expressed themselves willing to make these rings at a cost of five shillings per set of ten in a suitable case.

"In the course of etching the specimen, it is inadvisable to have it mounted on the slide, as when mounted it is difficult to remove the water used in washing from the irregular under-surface of the metal, and this, if left there, causes rusting.

"To temporarily hold the specimens in position, I have used with success a glass slide on which is cemented a short length of brass tube. This is nearly filled with sea or silver sand.

"The microscope being in a vertical position, the special slide, with its cylinder containing the sand, is placed on the stage. The 'object' is placed face upward on the sand, and a piece of glass plate pressed upon it till it meets the walls of the brass cylinder. The glass is then removed, leaving the specimen bedded in the sand with its polished surface parallel with the bed of the stage. After preliminary examination the section can be removed with a pair of forceps and further treated, or finally be mounted for storage.

"When a cover-glass is admissible, it is possible to cover up the polished surface in the usual way with Canada balsam, and many of my specimens mounted four years ago in that way are still quite bright.

Microscope Accessories.

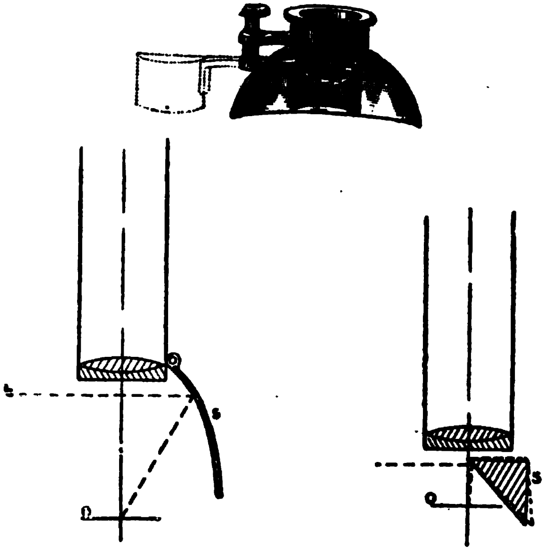
"It is only necessary to describe here those parts of the microscope outfit which have been specially designed for illuminating opaque objects, and for changing the object glasses.

"The systems for illumination may be divided into two main classes:—

"(*a*) For illuminating by light thrown on the object by reflectors fixed below the object glass, only admissible with low power objectives when there is ample distance between the object glass and the object.

"(*b*) For illuminating objects when using higher power objectives than about ¼-in. focal length; mirrors or prisms placed above the object glass are necessary.

"The combination sold by Messrs. Beck & Co.—known as the Sorby Beck Reflectors for oblique and vertical illumination —is the most useful reflector yet designed for low power objectives. The following illustrations will show the construction of this piece of apparatus.



Beck's Side Reflector. Sorby's Reflector.

Figs. 15, 16, 17.

L—Rays of incident light. S—Reflectors. O—Objects.

"When working with the parabolic reflector of Beck, the little silver mirror of Sorby with a face at 45° to the horizontal is thrown outside by turning the milled head A. The horizontal rays of light falling on the silvered parabola are reflected at various angles upon the object placed below, and if the object is a perfectly flat surface, the whole of the light will be thrown off, and none will enter the object glass; hence, such a flat surface cannot be seen on looking through the microscope; it appears quite black.

"The parabolic side reflector on this account is most useful in determining which part of a structure is horizontal and which broken up.

"It is useful also in determining when one constituent is in relief, for as the light falls on one side of the projecting portion it brightly illuminates it, leaving the other side in shade.

"The vertical illuminating mirror of Sorby when swung over the object partly eclipses the object glass, but the horizontal rays of light, falling on the mirror, reflect vertical rays upon the object, and these rays are again reflected into the microscope.

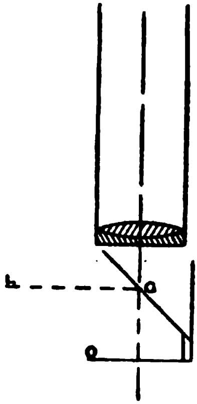
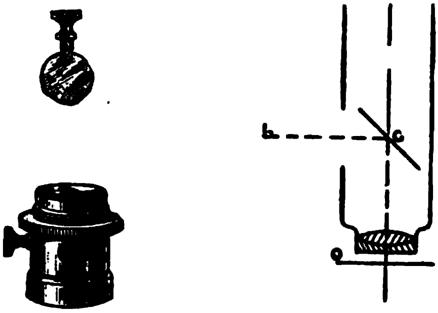


FIG. 18.
L—Light rays. G—Glass reflector. O—Object.

"It is not, however, necessary to use this special apparatus when only vertical illumination is required. A piece of blackened cardboard fixed behind the object in a vertical position, and a cover glass placed over the object at an angle of 45° gives excellent illumination. The card and cover glass can be temporarily attached to the object glass slip with plastic wax. It only takes one minute to arrange.

"The horizontal incident rays from the lamp are reflected downwards, on to the object, and thus brightly illuminates the specimen. The light from the object passes upwards through the reflector and into the object glass and microscope.

"(*b*) *Reflectors for High Powers.*



FIGS. 19, 20.
L—Light rays. G—Glass reflector. O—Object.

"The Beck illuminator for high power is similar to the last described, but it is placed inside of a special adaptor which is screwed into the microscope tube above the object glass. The light is admitted at the side of the adaptor through an opening which is capable of adjustment by a suitable diaphragm.

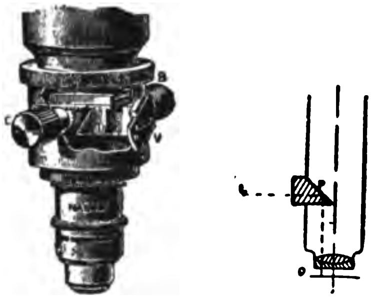
"The light falling on the cover glass is reflected downwards through the object glass on to the object. The rays from the object pass upwards again through the object glass and the reflector to the eye-piece.

"Professor Martens and others use a right-angled glass prism above the object glass, adjustable by a rack and screw so as to move it in or out in a horizontal direction. The light from the prism passes downwards in a vertical direction, illuminating the object most perfectly. Messrs. Nachet & Sons are makers of a most perfect illuminating device of this kind which, in my hands, has given most excellent results when photographing up to 500 diameters.

"It is constantly necessary in examining metal sections to view the structure under different object glasses, commencing with a low power, such as an inch objective fitted with a Sorby Beck reflector; next with a $\frac{1}{4}$-in. or $\frac{1}{8}$-in. objective with the Nachet prism illuminator; and finally with a $\frac{1}{8}$-in. or $\frac{1}{16}$-in. oil immersion objective with the Beck illuminator.

"For ordinary microscopic work, when it is not necessary to have the somewhat cumbersome illuminating auxiliary apparatus attached to the object glasses, the triple nose-piece is all that is required; but as the illuminators interfere with the free rotation of the nose-piece it is not suitable for metallographic work.

"Carl Zeiss, of Jena, has designed a much more useful changing apparatus, which is called by him an Object Glass Slide Changer.



FIGS. 21, 22.
L—Light rays. P—Prism. O—Object.

"A grooved slanting slide-holder is screwed into the microscopic tube, and each object glass is fitted with a slide-piece, which simply slides into position in the holder.

"The object glasses are adjusted by a little set screw to make the centre of each register in the same vertical line.

"Changing from one object glass to another does not take more than two or three seconds.

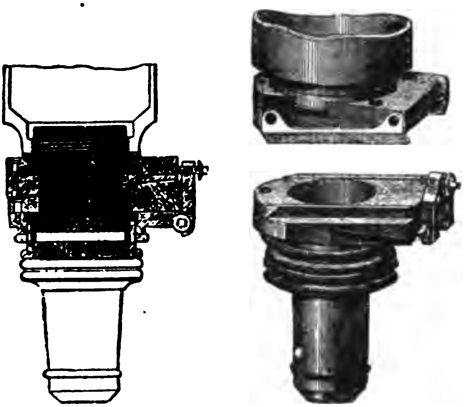
"The following block will explain itself.

"It is absolutely necessary to have command of a brilliant light, more particularly when the metal objects have to be photographed.

"If electricity is available either an arc lamp or a forced incandescent light is the best; failing electricity an incandescent Welsbach gas lamp, or an acetylene jet will answer well. I have used with success the Welsbach lamp for all my work.

"A bull's eye condenser should be placed between the light and the reflector, so as to concentrate the rays as much as possible.

"In many cases it is necessary to cut off the full force of the light by means of diaphragms or stops, especially when using the oil immersion high power objectives, and in all cases when the object has a brilliant reflecting surface.



FIGS. 23, 24, 25.

"This is easily done by using the stops or regulators supplied with the Beck and Nachet reflectors.

"The light may be more diffused by placing a piece of ground glass or thin tissue paper in front of the lamp.

"It is only by experience and practice one can find how best to illuminate an object.

Photographing the Object.

"Of cameras for photographing microscopic objects there are very many. Nearly all the manufacturers of microscopes also make cameras for this purpose. There are two systems of using these cameras. The first is that in which the microscope is turned over so that the tube is horizontal, and the eye-piece is fitted into the front of the camera which is also placed in a horizontal position. The second is that in which the microscope and camera are placed in a vertical position.

"In the first case, the microscope must be fitted with a mechanical stage, and the objects must be securely mounted on the glass slides. The long camera monopolises a considerable table area.

"In the second arrangement the microscope need not have a mechanical stage, and the objects need not be securely mounted, and very little table room is required. The microscope, moreover, being in a vertical position, it is much more convenient to examine the specimens before the camera is lowered over the eye-piece. There is only one disadvantage to this system, and that is the necessity of standing on a chair or stool to focus the object, but that is a very slight drawback.

"Altogether the advantages in favour of the vertical system are overwhelming.

"A very simple arrangement for photographing vertically can be made with the assistance of a joiner, at a small cost, not exceeding more than thirty shillings. Such an apparatus is described in the paper referred to previously.*

"A very convenient form of camera and stand is made by Messrs. Nachet & Sons, Paris. An illustration of this camera is shown in Fig. 26, the working of which will be understood with very little explanation.

"It will be noticed that the camera slides up and down on the rigid supports on each side of the vertical microscope. It can also be swung clear of the microscope, so that it does not come in contact with the head of the observer when using the instrument during visual examination.

"In taking the photograph after having obtained the particular field of the object in position, without removing the eye-piece, the camera is placed in position, the hood lowered over the eye-piece, and the box is raised to the point corresponding to the magnification required. A clear glass plate is substituted for the ground glass screen at the top of the camera. The focussing is effected by applying the eye to a No. 1 eye-piece placed on the centre of the glass, and by means of the micrometer screw adjustment is made till the object is seen most distinctly. The glass screen is removed, and a slide with the sensitive dry plate is fixed into place, the slide drawn, and the exposure made. The time of exposure depends on the brightness of the object, the quantity of light admitted, and on the magnification. In

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* *Iron and Steel Journal*, No. 1, 1897.

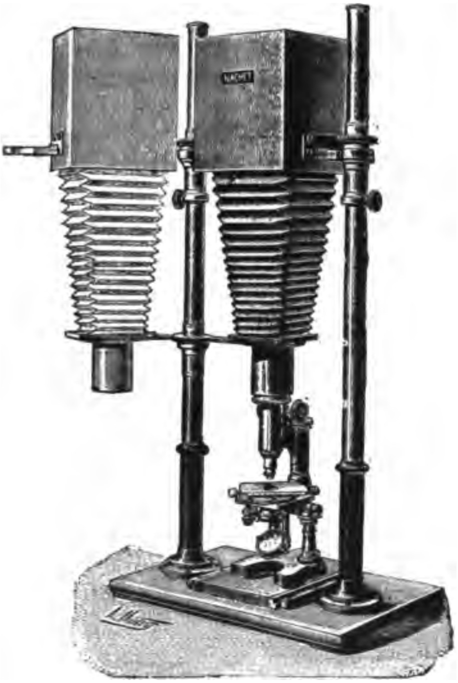

FIG. 25.

my own practice, using the Empress isochromatic plates (Ilford), when clear objects are used, and a magnification of 100 diameters photographed, from one to five minutes' exposure is necessary to get a good impression, but some objects take more than an hour. The development is done in the usual way, and it is not necessary to describe the process. Personally, I prefer the ready-made solution called "Rodenal," diluted with ten times its own volume of water.

"In every camera it is advisable to graduate one of the guides, and mark off the distance from the microscope stage to the back of the camera in inches or centimetres, and to prepare tables with each object glass correlating the graduation with the diameters magnified. This is done by placing a micrometer slide, 1 mm. divided into 100, on the stage, and allowing transmitted light to pass through it and the microscope, and to fall upon a ground glass screen, ruled with lines 1 centimetre apart, in the camera. The magnification of the divisions of the micrometer slide can be seen on the ground glass, and the comparison with the ruled lines thereon compared.

"By moving the camera box upwards and taking repeated observations, a table can be tabulated for each particular object glass and eye-piece employed. Allowance, of course, must be made for the difference in height of the various thicknesses of the sections on the stage."

Structure of Pig Iron.—Commenting upon the structure of phosphoric pig iron, in a communication to the Author, Mr. Stead says:—

"What is generally admitted is that in the greyest iron the structure appears to be ferrite cut up by plates of graphite, thin and numerous in close grained iron, and thick and more massive in the more open varieties. Analysis shows that in such iron there is practically no combined carbon, therefore practically no pearlite and no cementite is present.

"As the combined carbon increases, at first pearlite appears, and afterwards, when the quantity is considerable, massive cementite becomes evident in which the carbon exists in the combined state. In white iron the structure appears to be essentially massive cementite (Fe_3C) and pearlite (alternative layers of carbide of iron and ferrite).

"These statements, I think, will be admitted by all metallographers, who have made an elementary study of the structure of such irons as are made from Cumberland or Spanish iron ores.

"During the last twelve months I have made special examinations to ascertain if there was any material difference between the structure of phosphorettic and non-phosphorettic pig irons. The results obtained have proved that the difference is considerable.

"The phosphorus in phosphorettic pig irons exists as phosphide of iron in a eutectic as a definite chemical compound having the formula Fe_3P, and this compound exists in isolated segregated masses, easily identified in the grey irons but less so in white irons.

"Practical men know very well that it is impossible, from the appearance of the fracture of pig metal to tell whether a cast iron is phosphorettic or not.

"By polishing a chip of the iron and etching it with iodine solution, or by heat tinting, it is now quite easy to see under the microscope the phosphide areas and to make an approximate estimate of the quantity of phosphorus present.

"A series of specimens of grey iron containing 1·5 per cent., 1·00 per cent., 0·50 per cent., 0·25 per cent., 0·10 per cent., and 0·05 per cent. phosphorus were prepared. After polishing they were mounted. The slides were not marked, so that it was impossible to tell which was which; yet it was easy to arrange them in sequential order on examination under the microscope.

"It was also possible to detect isolated specks of phosphide in slowly cooled grey metal containing less than 0·05 per cent. phosphorus.

"The portions last to solidify were those which contained the most phosphorus, and, as one would expect, the phosphide areas were always found in those positions, where, when solidifying, the first portions to freeze forced them. They were generally found farthest removed from the plates of graphite in grey iron, the graphite representing the portions first to fall out of solution.

" The phosphide in white irons occupied areas between the massive plates of cementite, which latter first crystallised, leaving the fusible phosphorus eutectic compound to solidify in those areas whither it was driven by the first constituents which fell out of solution.

" In any of the ordinary Cleveland white pig irons it is easy to identify the massive carbide plates, the pearly constituent of Dr. Sorby (Perlite, Pearlite, Pearlyte), the second pearly constituent or eutectic of phosphorus, and also the sulphides.

" The best way of treating a specimen to enable one to identify these constituents is to heat the polished metal upon a hot plate till it assumes a purple tint to the naked eye. The various constituents oxidise at different rates, and assume corresponding coloured films. Heated in this way, pearlite assumes a blue tint with brown-red worm-like layers of carbide of iron ; the massive carbide takes a fine red-brown tint; the phosphide of iron a pale yellow colour.

" The sulphides do not apparently change colour, but are easily detected, as they appear of a slate colour and exist in isolated segregations, very often in idiomorphic crystals.

" These colours only appear in this order if the heating has been correctly done. By longer heating the colours advance in sequentially progressive steps forward through yellow, brown, orange, red, purple, blue, to slate grey.

" Such specimens properly heat tinted, are magnificent microscopic objects.

" The ordinary methods of etching do not enable one to find any difference between carbides and phosphides, and in the past some metallographers, myself included, have mistaken the one for the other.

" The heat tinting process enabled them to detect with certainty the phosphides.

" The phosphorus eutectics of white iron are quite different from those of grey pig, both in form and composition."

Description of Photographs.[*]

[See illustrations following p. 61*y*. All specimens were illuminated by vertical light.]

Photograph No. I.—This is a specimen of Cleveland iron, No. 3, " Cargo Fleet " brand, showing sections of the graphite plates (black) ferrite constituting the ground mass, and the phosphide eutectic in irregular patches of mottled appearance, distributed at intervals over the whole area, magnified about 50 diameters.

Photograph No. II.—This specimen is the same as No. I., but magnified 250 diameters. It will be observed that the compound character of the eutectic is very evident.

Photograph No. III.—This is a highly magnified photograph of one of the phosphide eutectic areas, showing its characteristic duplex nature. The ground mass was darkened to more clearly show the structure. This was magnified 400 diameters.

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[*] Photographs Nos. I. to VII. appeared as illustrations to a Paper by Mr. Stead, published in the " Proceedings " of the Cleveland Institution of Engineers (February 26, 1900); Nos. VIII. and IX. were issued subsequently.

Photograph No. IV.—Cleveland iron—Clarence white iron. The structure was developed by etching with iodine, which had no action on the phosphide or carbide. The continuous white areas consist of massive carbide. The mottled area the phosphide eutectic, consisting in the dark spots of pearlyte, and the white parts of juxapose, carbide and phosphide of iron. The dark irregular shaped masses are pearlyte, in which the plates of carbide of iron are very thick. Magnified 250 diameters.

Note that the photograph gives no indication where the carbide and phosphide join.

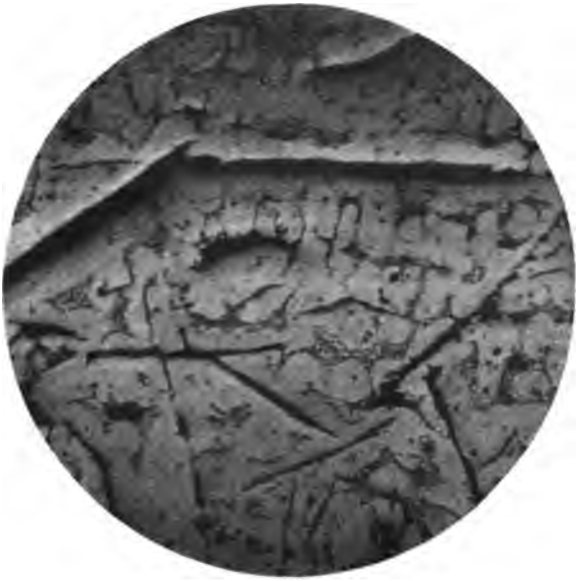
Photograph No. V.—Basic iron, containing 2·9 per cent. of phosphorus. The section was not etched with iodine, but its structure was developed by the "heat tinting" process. The flat dark area coloured red in the specimen, but shown nearly black in the photograph, is the massive carbide of iron corresponding to the broad white areas in the previous photograph. The irregular globular-shaped masses, nearly white, in the photograph sky blue, are pearlyte. In the specimen the carbide was red, the phosphide pale yellow, and the pearlyte blue. Note the clearly marked division between the massive carbide and the phosphorus eutectic, so different from what is seen in the iodine etched specimen of No. 4. Magnified 200 diameters.

Photograph No. VI.—Iron containing 6 per cent. phosphorus and 1·6 per cent. carbon, etched with iodine. The white bands are sections of carbide of iron which first fell out of solution on solidifying. The broken up structure is the phosphide eutectic constituting now, in consequence of the large proportion of phosphorus, the ground mass. The dark areas are pearlyte. Magnified 100 diameters.

Photograph No. VII.—White hematite pig iron containing 0·04 per cent. phosphorus, etched with iodine. The dark areas are pearlyte, the white parts carbide of iron. No phosphide eutectic is shown in this photograph. The small half-tone areas seen embedded in the cementite are mainly sulphide of manganese. Magnified 100 diameters.

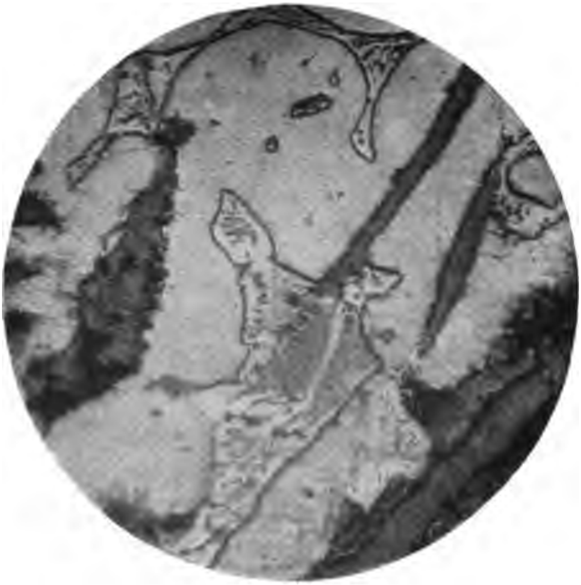
Photograph No. VIII.—Pearlyte in steel. Magnified 350 diameters.

Photograph No. IX.—Ingot containing 11·10 per cent. phosphorus and 88·9 per cent. iron, showing sections of rhombic or oblique idiomorphic crystals of Fe_3P embedded in a ground mass of the eutectic. Magnified 60 diameters.

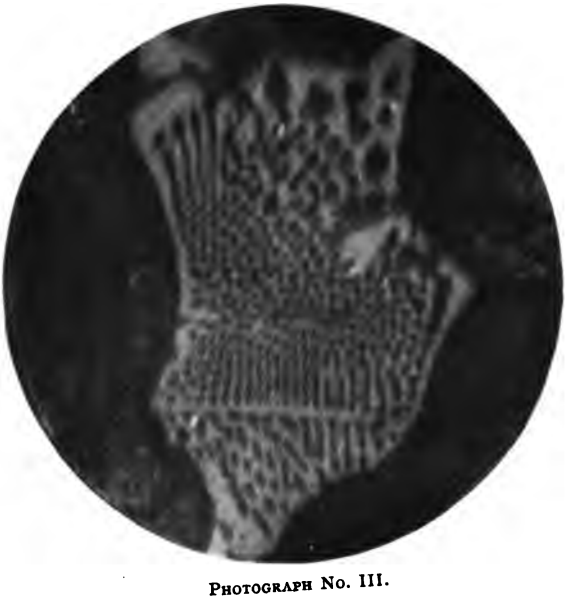


PHOTOGRAPH No. I.

To face p. 61y.

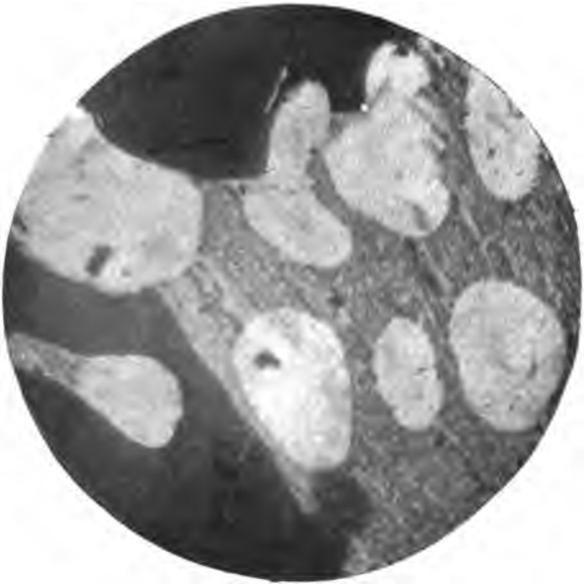


Photograph No. II.

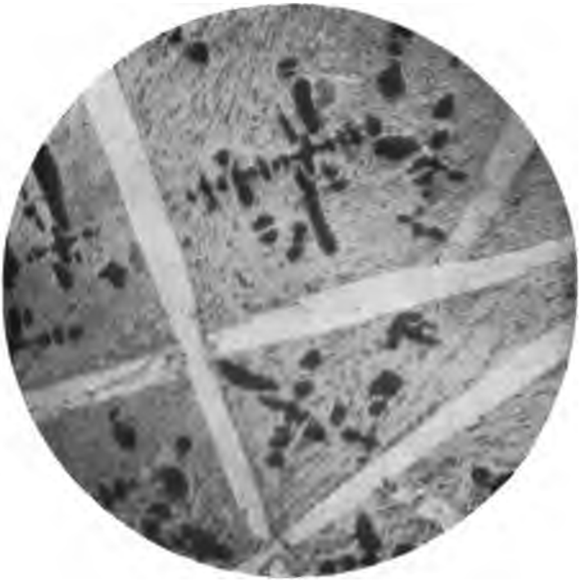


PHOTOGRAPH No. III.

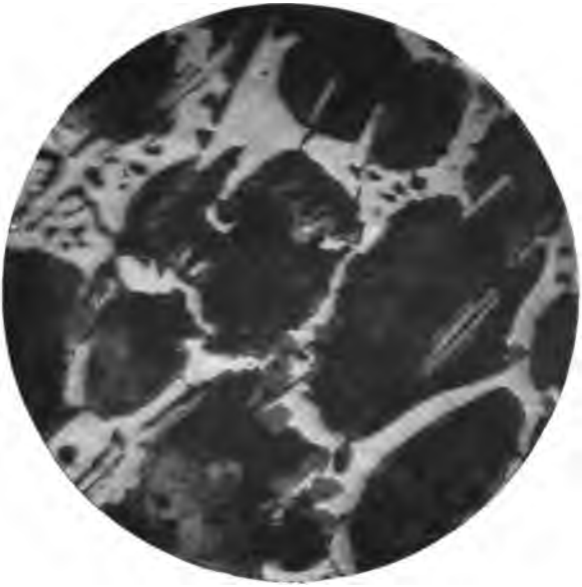
Photograph No. IV.



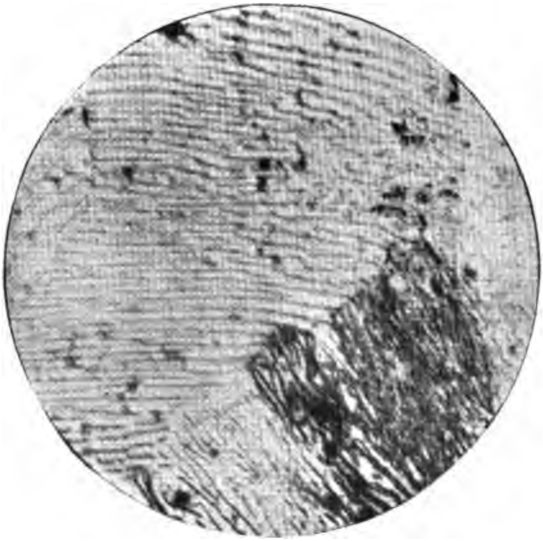
Photograph No. V.



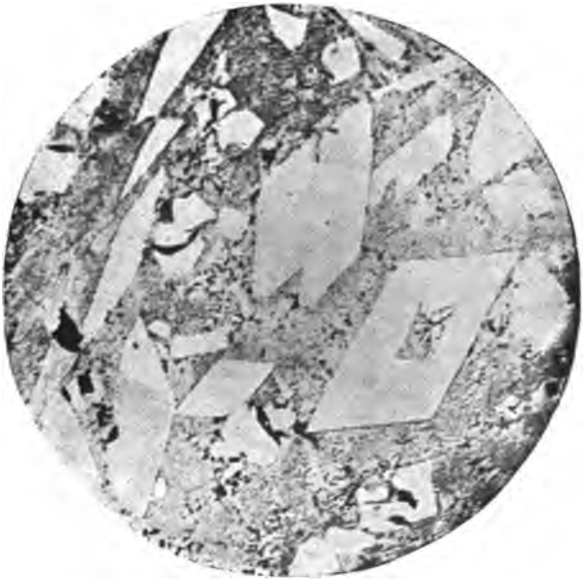
Photograph No. VI.



Photograph No. VII.



Photograph No. VIII.



Photograph No. IX.

COPPER.

This important metal (it is hardly necessary to say) is now almost indispensable to the requirements of the engineer, being largely used for locomotive fire-boxes, tubes, and numerous other purposes. It is also a constituent of a great number of alloys.

Copper when pure has a red colour, is ductile, tough, and highly malleable, and is one of the best conductors of heat and electricity known; it has a specific gravity of 8·96, and melts at about 1,100° C. Owing to the complex nature of the ores from which copper is extracted, the number of impurities it may contain are many; and since very small quantities of some of these impurities render copper practically useless for some purposes, it becomes very important that in buying it should be ascertained that the metal is of the desired quality. The impurities that may be expected to be found in copper are arsenic, antimony, lead, bismuth, sulphur, oxygen, nickel, tin, iron, zinc.

Arsenic, antimony, and lead in small quantities have a very injurious effect upon copper, making the metal brittle and "cold-short," although lead is added for rolling purposes to the extent of $\frac{1}{2}$ per cent., and the presence of arsenic in limited quantities appears to benefit the metal for certain purposes. Bismuth and sulphur are very undesirable elements in copper. Bismuth present to the extent of only $\frac{1}{200}$ is liable to make the metal "cold-short." Silver generally occurs in copper in such small quantities as not to affect its quality. Nickel and iron in small quantities render copper less malleable, more infusible and brittle. Oxygen present as cuprous oxide plays an important part in the metallurgy of copper. Being soluble in the molten metal it makes it hard and brittle; it is, however, for the most part eliminated from the metal while in the molten state by a process termed poling, which consists in covering the metal with powdered coke or anthracite and stirring with a pole of green wood, by which means the oxide is reduced to the metallic state. Now, although oxygen in itself produces a brittle metal, it has the desirable property—when present in certain proportions—of neutralizing the injurious effects of other impurities present; so that it is essential that the poling of commercial copper should not be carried on as far as to allow the prejudicial effects of the metallic impurities to predominate.

The principal ores from which copper is smelted are the *red* and *black oxides* found in Cornwall, Australia, and South America; *copper pyrites* found in Cornwall, Australia, Cuba, Siberia, and Sweden; *green* and *blue carbonates* found in America, Australia, and Siberia; and *grey ore*, being a mixture of the sulphides of copper, arsenic, antimony, and iron, occurring in Cornwall and Saxony. *Native copper* is found disseminated in granite in Wales and Cornwall, while on the shore of Lake Superior blocks of metallic crystallized copper, weighing as much as 400 tons, have been found.

The metallurgy of copper, owing to the variable and complex nature of its ores, is very complicated. The centre of the copper smelting industry in this country is at Swansea.

It is interesting to note (as pointed out by a recent writer*) the "striking difference in a modern copper smelting-plant when compared with those which were in operation twenty or thirty years ago. A 30 to 50 tons cupola was then considered a large furnace, whereas, if the notable copper mines of to-day are passed in review, it will be found that the up-to-date water-jackets are smelting, year in and year out, from 200 to 250 tons, and even 300 tons daily. The same comparison applies to reverberatory-smelting furnaces, whose ancestors can be seen in Swansea, where tradition and custom have adhered to the original size of 9 feet by 15 feet internal dimensions, whereas a modern reverberatory furnace has a laboratory of 16 feet by 35 feet, smelting 50 tons of ore daily; and even these are small when compared with those lately erected at the Boston and Montana Company's works, where regenerating reverberatory furnaces are smelting from 125 to 150 tons of roasted ore into matte daily."

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* Mr. M. Eissler, in a Paper on the Production of Copper, read before the Institution of Mining Engineers on May 23rd, 1901. See also the same Author's work on "The Hydro-Metallurgy of Copper Ores," now (December, 1901) in the press (London; Crosby Lockwood & Son).

"The most notable advance in smelting," says the same writer, "has been the utilisation of the sulphur, iron, and other oxidizable substances in the ores as generators of heat, so that they practically smelt themselves, without the aid of extraneous heat. The application of this principle is known as 'pyritic smelting.'

"All copper, however, is not produced by smelting: in some isolated cases, as at Rio Tinto, in Spain, the copper contents are extracted by wet methods or leaching; and in Arizona, U.S.A,, sulphuric acid is used as a solvent for the extraction of copper. The brilliant results achieved at the Rio Tinto mines, by the natural decomposition of the ores, which after being exposed in immense heaps to atmospheric agencies, are then lixiviated, goes to prove that when local conditions favour the introduction of wet methods, copper can be won at a very low cost by these means."

Method for the Complete Analysis of Copper.

Insoluble Residue and Stock Solution.—Weigh out 50 grms. of the sample in turnings, and transfer to a 1¼ litre beaker, pour on 600 cc. of nitric acid, sp. gr. 1·2 = 6·15 E, and allow to digest on a warm plate until all that is soluble is dissolved; evaporate now over water bath until crystals of copper nitrate come out. Dissolve in 800 cc. of distilled water and filter off any residue through a small tared filter paper; detach any portion adhering to the sides of the beaker with the aid of a feather and wash till free from the copper salt with E HNO_3 and finally with water; dry the filter at 100 C., and weigh between watch glasses. If the residue is at all considerable it is to be kept and fused with sodic sulphide, and treated by processes herein described. There will be a separate method adopted for the estimation of tin. The filtrate is now diluted to 1,000 cc. at 15·5° C., and poured into a dry stoppered bottle, from which known volumes are to be taken for the estimation of most of the impurities.

Estimation of Fe, Ni, Co, Zn.—200 cc. (equivalent to 10 grms. of sample) of the stock solution, are measured out accurately, and poured into a litre beaker; add now 20 cc. of sulphuric acid, sp. gr. 1·84 = 36 E, and evaporate as far as possible on the water bath and finally on the sand bath until all fumes of H_2SO_4 are eliminated. Allow to cool and digest with 300 cc. of $\frac{E}{2}$ H_2SO_4, until all that is soluble is dissolved, decant as much as possible of the clear blue solution into a tared 1½ litre flask, provided with a cork, and pass the residual solution containing any $PbSO_4$, &c., through a filter paper and finally wash with $\frac{E}{2}$ H_2SO_4 until free from copper salt—reject residue. To the solution add 50 cc. of strong HCl = 10 E, and dilute to 1,000 cc., the solution being now ·65 E total free acid. Heat the solution to 70° C. and saturate with sulphuretted hydrogen, after which the cork is introduced, allowed to cool to ordinary temperature, and the whole weighed. Deduct from this the weight of the dried flask, + 15·07 grms. CuS (the amount of CuS formed by the 10 grms. of sample taken; this is not strictly speaking correct, but there is no practical error introduced for ordinary coppers) and the amount of liquid present will be obtained. Decant through a filter carefully and as quickly as possible (to prevent oxidation) into another tared 1½ litre flask and weigh; and calculate from this the amount of sample equivalent to the volume decanted (*vide* example, page 76). The liquid is now poured out of the flask into a beaker and evaporated to complete dryness over the sand bath. Dissolve the residue in 20 cc. of ½ E HCl, pour into a small, tall beaker and saturate with SH_2, and filter off any little CuS, &c., that was not completely separated in the first instance. Neutralize the solution with 20 E AmHO, add ½ cc. in excess, and saturate with SH_2. Allow to settle in a warm place for about a quarter of an hour and filter off the precipitated sulphides of Fe, Ni, &c.; wash quickly with ½ E Am_2S solution and dissolve residue in 3 cc. warm 5 E HCl, with addition of a crystal of chlorate of potash: boil the solution carefully until all free chlorine has escaped, add 4 cc. of 5 E AmCl, dilute to about 20 cc., and add, very cautiously, 5 E solution of ammonic carbonate, until nearly all the free acid is neutralized; then add, drop by drop, a solution of E ammonic carbonate, until a slight turbidity is produced. Heat slowly to boiling, cool, and add 3 cc. of 5 E AmHO; allow to stand a short time, filter through a small filter paper, wash with ½ E AmCl, reserve filtrate, re-dissolve in 3 cc. of 5 E HCl, and re-precipitate the iron now as ferric hydrate [$Fe_2(HO)_6$] with 1 cc. of ammonic hydrate ·880 sp. gr. = 20 E; heat to boiling, and filter off through one of Schleicher & Schüll's smallest size chemically pure filter papers, leaving less than ·0001 grm. of ash on incineration, a quantity which may be disregarded in the determination. (It may be here remarked that throughout the copper analysis, where such small quantities are to be weighed, it will be advisable to use these papers. They can be obtained from Messrs. Townson & Mercer, London, who are the sole agents.) Wash the precipitate with water, until the washings no longer give a turbidity with nitrate of silver, mix filtrate and reserve, spread the filter on to a watch glass and dry in the water oven. Scrape as much as possible of the oxide into a small tared porcelain crucible and ignite the filter paper separately, adding the residue to the main portion. The crucible and its contents are now gradually ignited to a full red heat in the oxidizing flame of a Bunsen or in a good muffle, after which it is cooled in a good desiccator and weighed. The increase in weight of the crucible is the amount of iron originally present in 10 grms. of the sample now in the state of peroxide (Fe_2O_3). This weight, multiplied by ·700, and divided by amount of sample taken, × 100, will give the percentage of iron in the sample.

The mixed filtrates, containing the zinc, &c., are now evaporated to 30 cc. and made just acid with HCl, and then *just* alkaline with E Na_2CO_3; add 4 drops of 5 E HCl, saturate with SH_2, filter off any zinc sulphide if present, and wash. Boil the solution until SH_2 has gone off, neutralize with

E Na_2CO_3, make $\frac{E}{5}$ with acetic acid, add 2 cc. E acetate of soda, heat to 70° C., saturate with SH_2, allow precipitate to subside, filter off the nickel sulphide quickly, and wash with SH_2 water (test the solution for manganese by neutralizing with 20 E AmHO, and adding 5 cc. 5 E Am_2S). Dissolve off the filter with 5 cc. of 10 E HCl, with addition of a crystal of potassic chlorate, boil till chlorous odour has gone, and wash out into a porcelain dish; volume being here now 15 cc., neutralize with 2 E Na_2CO_3, boil, and add 2 cc. of 2 E NaHO solution (pure) to the hot solution, and boil for a few minutes: filter off hydrate of nickel, wash with boiling water till free from alkali, dry in water oven, scrape precipitate into a tared crucible, ignite the filter separately, and add residue to main portion: ignite the crucible at a red heat for ten minutes, cool and weigh the NiO, which $\times$ ·7867 and $\div$ the amount of sample taken, $\times$ 100 = percentage of nickel in the sample.

Test the nickel oxide for cobalt by treating the residue in the crucible with ½ cc. of aqua regia, evaporating to dryness, dissolving in 5 cc. of water, adding a drop of 5 E acetic acid and 2 cc. of 4 E potassic nitrite solution, and allowing to stand at 50° C. for some hours, when, if cobalt is present, a yellow precipitate of double nitrite of cobalt and potassium will be produced.

Estimation of Antimony and Arsenic (*Abel's process*).—Take 200 cc. of stock solution, equivalent to 10 grms. of original sample, and pour into a litre beaker. Add 5 cc. of an E solution of nitrate of lead [$Pb(NO_3)_2$], 70 cc. of 5 E AmHO, and 10 cc. 5 E Am_2CO_3. Dilute to 800 cc., and allow to stand twenty-four hours with frequent stirring, after which it is allowed to stand without stirring, until the precipitate of arseniate, antimoniate, carbonate, &c., of lead has settled to the bottom. Decant as much as possible of the clear solution, filter off the remaining solution, wash the precipitate with 2 E AmHO till free from copper salt, &c., spread out the filter on a glass plate, carefully remove as much as possible of the precipitate into an 80 cc. beaker with a platinum spatula, and remove any precipitate adhering to the paper with 30 cc. of 1½ E oxalic acid, with the aid of a wash bottle. Boil the solution for half an hour, filter off, and wash with water. The antimony and arsenic is now in solution. Dilute filtrate to 150 cc., neutralize with 2 E pure solution of sodic hydrate, add 7 grms. by weight in excess, saturate with sulphuretted hydrogen (SH_2), allow to stand in a warm place for some time, filter off sulphide of lead, &c., and wash with ½ E Na_2S solution. Neglect residue, dilute filtrate to 225 cc., carefully add 20 cc. of 10 E HCl, allow the precipitate of antimony and arsenic sulphides, and free sulphur, to settle in a warm place for a couple of hours, filter off, wash with SH_2 water, dry in water oven, half fill a small tall beaker with bisulphide of carbon, fold the paper up with its contents, place it cone downwards into the CS_2, and cover with a watch glass; by this means the great bulk of the free sulphur is dissolved out.

After digesting for a couple of hours the filter is spread out on a watch glass and dried, the precipitate brushed into a small beaker and dissolved in a mixture of 2 cc. 22 E HNO_3 (fuming) and 6 cc. 10 E HCl at a gentle heat. Dilute with water to 50 cc., add 4 grms. of tartaric acid previously dissolved in 5 cc. of water, neutralize with 5 E AmHO and add 4 cc. in excess. Add 10 cc. of E magnesia mixture (prepared by dissolving 68 grms. magnesic chloride and 165 grms. AmCl in 290 cc. of 5 E AmHO and diluting to 1 litre) and dilute to 100 cc., stir well and allow to stand twenty-four hours, filter off the arseniate of magnesia and ammonia, detaching the last portions of the precipitate from the beaker with the aid of some of the filtrate and a feather. This is adopted in order to minimize the number of washings with the 2 E AmHO in which the precipitate is somewhat soluble. Wash with 2 E AmHO, and note the total volume of the solution, which should be about 120 cc., and reserve. Dry the precipitate in the water oven, brush into a tared porcelain or platinum crucible, saturate the paper with E ammonium nitrate solution, dry, ignite by means of platinum wire and Bunsen, and add the residue to mean portion. Ignite *gradually*, until the crucible is at a bright red heat, cool, and weigh. The increase of weight of the crucible is due to pyroarsenate of magnesia ($Mg_2As_2O_7$). Add to this weight ·0032 grm. due to the solubility of the arseniate of magnesia and ammonia in the 120 cc. of the ammoniacal solution, multiply this weight by ·4839 × 10, and this will give the percentage of arsenic in the sample.

The filtrate containing the antimony is now neutralized with 10 E HCl and 5 cc. added in excess. Saturate with SH_2, allow the sulphide of antimony to settle in a warm place, filter off, wash with SH_2 water, dry in water oven, scrape off paper into a small tared crucible, moisten the paper with E $AmNO_3$ solution, dry and ignite, and add residue to crucible; add 8 to 10 times its bulk of 22 E HNO_3, cover with a watch glass and digest for some time at a gentle heat; evaporate off excess of acid and carefully ignite at a red heat for ten minutes, cool in desiccator and weigh the Sb_2O_4. Multiply this weight by ·7922 × 10, and this will give the percentage of Sb present in sample.

Estimation of Lead and Bismuth (*Abel's Process*).—Take 200 cc. of the stock solution (= 10 grms. of sample) and pour into a litre beaker. Now add 20 cc. of E sodic phosphate solution, dilute to 650 cc., add 60 cc., 20 E AmHO, stir well, and allow to stand for forty-eight hours. Decant as much as possible of the clear liquor, filter the residue on a small filter paper, wash till free from copper salts with 2 E AmHO, and dissolve the phosphates of lead, bismuth, &c., on the filter into a small beaker with 5 cc. of warm 10 E HCl, washing out with hot water. Let volume here be now 20 cc., neutralize with 20 E AmHO, add 1 cc. in excess, saturate with SH_2 gas, allow the precipitated sulphides of lead, bismuth, &c., to subside in a warm place, filter and wash with SH_2 water, place a test tube under the funnel and pour on to the precipitate 5 cc. of 8 E HNO_3 (warm), passing it through several times.

There are invariably lumps of dark sulphur containing traces of lead and bismuth left on the paper. To separate the sulphur, push the sulphur particles as far as possible down to the bottom of the filter, dry in water oven, and digest in some carbon bisulphide contained in a small beaker until the sulphur is dissolved out; now dry, treat with 8 E HNO_3, and add this to the main portion. The filter is dried, moistened with ammonium nitrate, carefully incinerated, and the ash likewise added to main solution. The whole is boiled until all is dissolved. Dilute to 15 cc., nearly neutralize solution with AmHO, using 20 E at first, and towards the end a 5 E solution, drop by drop, until the precipitate which is formed goes into solution with difficulty, and, the solution being perfectly clear, add now some recently precipitated copper hydrate, $Cu(HO)_2$, in slight excess with the aid of a glass spatula, stir well, and allow to stand a couple of hours with frequent stirring. (The copper hydrate is prepared by diluting 5 cc. of E $CuCl_2$ to 80 cc., and adding, gradually stirring, 15 cc. of $\frac{E}{2}$ NaHO solution, in the cold, filtering, and washing with water till free from NaHO.) This quantity of $Cu(HO)_2$ will generally be found sufficient for ordinary coppers. The bismuth is thus precipitated as $Bi(HO)_3$, and the lead left in solution as nitrate. Filter off and wash with hot water, and reserve filtrate for the estimation of lead. Dissolve off the $Bi(HO)_3$ and excess of $Cu(HO)_2$ in 3 cc. of 5 E HNO_3, wash the paper with $\frac{E}{5}$ HNO_3, dilute to 20 cc, saturate with SH_2, allow to settle, filter off sulphides, dissolve in 8 E HNO_3 as before, dilute to 15 cc., neutralize with 5 E Am_2CO_3 add 5 cc. in excess, and allow to digest on a hot plate for some time; filter off the bismuth carbonate, wash, dry in water oven, brush off to a small watch glass, ignite the filter carefully, and add the ash to a tared crucible. Digest this in a few drops of 16 E HNO_3, evaporate, add main portion of precipitate from watch glass, and ignite the whole cautiously in the oxidizing flame of a Bunsen burner for ten minutes, cool and weigh. Increase in weight $= Bi_2O_3 \times \cdot 8965 \times 10 =$ percentage of bismuth present in sample.

[This process is liable to give low results for lead. The lead could be estimated in the portion taken for the iron, &c., estimation, but the residue should be taken up in 5 E sulphuric acid, to take bismuth sulphate in solution, and then diluted five times with water before filtering off the lead sulphate.]

Estimation of Combined Oxygen (*Abel's Process*).—Clean a piece of the sample 2 in. × 1½ in. × ⅛ in. by immersing for a few seconds in 16 E HNO_3; wash with distilled water, then with alcohol, dry quickly in water oven and weigh. Immerse in 150 cc. of E $AgNO_3$ solution for three hours, occasionally scraping off the metallic silver, &c., from the surface of the copper with a stir rod. (Reaction with regard to the combined oxygen = $Cu_2O + 2\ AgNO_3 = Ag_2 + Cu(NO_3)_2CuO$.) The portion of the sample unacted upon is taken out and washed with the aid of a jet of water from a wash bottle, and a feather; the water is taken off with alcohol, and the sample dried in water oven, cooled, and weighed. The difference between this and the original weight is the amount of sample taken for the estimation.

The liquid and washings are decanted through a filter paper, and the precipitated silver and basic nitrate of copper washed with water, first six times by decantation combined with filtration, and finally filtered through the filter paper and washed till free from copper. The filter and its contents are now spread out on a sheet of glass and the precipitate transferred by means of a platinum spatula to an 80 cc. beaker. Now add 5 cc. of E $AgNO_3$ solution, digest for half-an-hour (this to ensure perfect reaction), add 25 cc. of *standard* $\frac{E}{4}$ H_2SO_4, allow to digest in an air oven for one hour at 50° C., filter by decantation, and wash free from acid. The reaction which takes place here is as follows: $Cu(NO_3)_2CuO + H_2SO_4 = Cu_2(NO_3)_2 + CuSO_4 + H_2O$, so that if we ascertain how much acid the basic nitrate has taken up, we can calculate its equivalent of oxygen. In order to ascertain this, fill a burette with *standard* $\frac{E}{4}$ Na_2CO_3, and run it gradually into the solution, which should be boiling (this is to eliminate CO_2 produced, which would keep any basic carbonate of Ag or Cu in solution) until a faint permanent precipitate is produced. Subtract the volume required from 25 cc. and this will give the amount of $\frac{E}{4}$ H_2SO_4 neutralized.

Now 1 cc. of $\frac{E}{4}$ H_2SO_4 = ·002 grm. of oxygen, and in order to get the percentage of oxygen present multiply the number of cc.'s of $\frac{E}{4}$ H_2SO_4 required by ·002, divide by the amount of sample taken, and multiply by 100.

Hampe's Method for Determining Oxygen in Copper.*—This has been found to be a more satisfactory method than Abel's. It consists in the extraction of the oxygen by ignition of the sample in pure hydrogen gas. There are various precautions necessary. The *modus operandi* is as follows:—

Thirty to forty grams of the copper are weighed for the test.

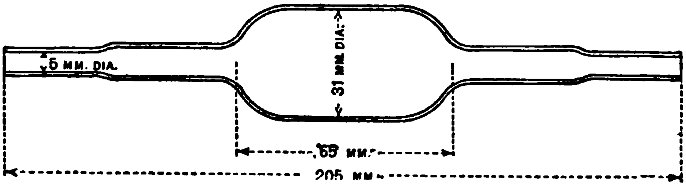

Fig. 27.

The sample should be in the form of bright turnings small enough to pass through a tube ¼-in. in diameter. The turnings must be first washed several times by decantation with ether to remove any grease, then dried in a water oven, and cooled in a desiccator. They are then transferred into a clean, dry, combustion bulb tube made of Jena glass of the shape and dimensions shown in Fig. 27.

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* *Vide Zei's. für Anal. Chem.*, 1874, pp. 176-234; and paper by L. Archbutt in the *Analyst*, October, 1900.

They weigh about 50 grams. The simplest way of getting the turnings into the tube is to cork one end, and pour the turnings through a funnel placed in the other end. After the cork and funnel are detached, the tube and its contents are accurately weighed. The weight of the dry empty tube having been previously ascertained, the exact weight of the copper is thus obtained. The tube and its contents is now supported on a sheet-iron tray lined with asbestos millboard, a piece of perforated sheet asbestos is bent over the top and kept in

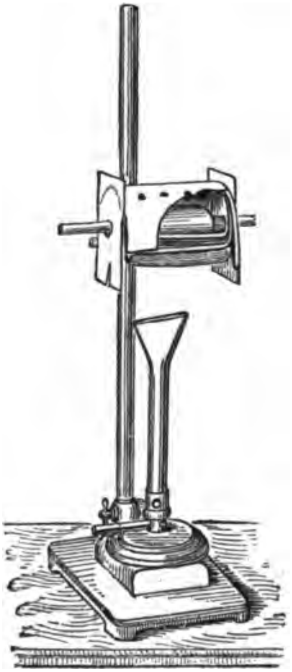

FIG. 28.

shape by an iron-wire frame, and flat pieces of asbestos, perforated in the centre to receive the ends of the bulb tube, are placed at the sides; the arrangement is seen in Fig. 28. The tube is heated by a Bunsen burner giving a broad, flat flame.

The tube is now ready to be attached to hydrogen generating and purifying apparatus, &c. The whole apparatus as used by Mr. Archbutt is seen in Fig. 29, and consists of—(1) Kipp generator; (2) Washing bottle containing caustic soda solution; (3) Tower containing stick caustic soda; (4) U-tube, containing re-fused dry caustic potash in small pieces; (5) Bulb-tube containing the prepared dry copper; (6) U-tube containing strong sulphuric acid; (7) Pipettes for measuring the rate of flow of hydrogen.

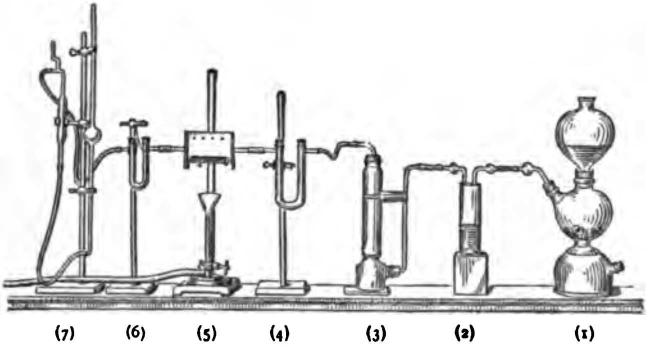

FIG. 29.

The apparatus having been all joined up, a current of hydrogen is passed through the apparatus at the rate of about 1 litre per hour, until the issuing gas burns quietly, which usually takes about fifteen minutes. The current of gas is then reduced about one-half, and if much arsenic is present in the copper it is further reduced; the copper is then heated up, with the flame just touching the iron support, until water begins to condense beyond the bulb tube.

Should there be a sublimate of As_4O_6 observed, the current of hydrogen is made very slow, and the tube is now more strongly heated so as to reduce the oxide to a mirror of metallic arsenic within the tube. Any water condensing between the bulb and the U-tube is driven along by the application of a small Bunsen flame. The tube containing the copper is now gradually heated to full redness, and when no more water condenses, the rate of hydrogen passing over is increased to 1 litre per hour. After the heat is thus kept on for about an hour, the gas is turned out, and the current of hydrogen meanwhile kept going until the tube is quite cold. The bulb-tube is now disconnected, and the hydrogen therein displaced by aspirating a current of dry air through the tube; the latter is then weighed, and the loss in the oxygen contained in the weight of copper taken, calculated in percentage.

It is a very satisfactory element in the process that the amount of oxygen in the copper taken by the loss in weight when heated in hydrogen is practically identical with that obtained by the direct weighing of the oxygen evolved as water.

The following is the result ot one of the many experiments made by Mr. Archbutt to prove the accuracy of the process, which is now beyond all doubt:—

SAMPLE OF COPPER FIREBOX PLATE.

	Grams.
Weight of copper taken	32·9989
" " " and tube before heating	60·6767
" " " " " after heating in CO_2	60·6745
" " " " " " " " hydrogen	60·6305
Loss of weight in hydrogen	·0440
Water absorption tube before experiment	49·4910
" " " after "	49·4427
Weight of water formed	·0483
Containing oxygen	·0429

Thus the percentage of oxygen found was:—

(1) By loss of weight of copper	0·133
(2) By weight of oxygen in water formed . . .	0·135

The variation of oxygen in samples of copper is considerable, and in conjunction with the proportion of other elements present it exercises an important influence on the physical properties of the metal. Out of thirty-three samples of rod, tube, and plate copper examined by the process, the amount of oxygen varied from 0·005 per cent. to 0·207 per cent.

Estimation of Silver and Sulphur.—Take 200 cc. of stock solution = 10 grms. of sample, and dilute to 700 cc. with distilled water. Heat to 70° C., add 2 cc. of 5 E HCl, allow the chloride of silver to settle, out of contact with light, for twenty-four hours, decant as much as possible of the clear fluid, filter off the AgCl on a small filter paper (chemically pure), wash with $\frac{E}{5}$ HNO_3 and finally with water till free from copper salt. Dry in water oven, scrape off into a tared porcelain crucible, ignite filter separately, and add residue to main portion; moisten with a couple of drops of 16 E HNO_3, to oxidize any reduced silver, add a drop of 10 E HCl to convert it into chloride, evaporate and carefully ignite at a dull red heat for ten minutes (do not fuse), cool in desiccator, and weigh. The weight of AgCl thus ascertained, multiplied by ·7527 × 100 = percentage of silver present in sample.

The filtrate containing the sulphur is now evaporated to dryness with 50 cc. of 10 E HCl. The residue is taken up in 50 cc. of water, and evaporated to dryness again with 50 cc. of 10 E HCl (this is to convert the copper into chloride, as the writer has found that $BaSO_4$ is soluble to an appreciable extent in a strong solution of nitrate of copper). Dissolve the residue in 200 cc. of $\frac{E}{10}$ HCl, and filter if necessary; boil, add 10 cc. of

E baric chloride solution, and allow to stand twenty-four hours; decant as much as possible of the clear solution, filter off the $BaSO_4$ on a small filter, wash with $\frac{E}{5}$ HCl, and finally with water until free from copper salts. Dry in water oven, brush off the precipitate into a tared crucible, ignite the filter separately, add residue to former, and ignite in oxidizing flame of Bunsen at a dull red heat for ten minutes. Allow to cool, and weigh.

The weight of $BaSO_4$ multiplied by ·1373 × 10 = the percentage of sulphur present in the sample.

Estimation of Tin.—Weigh out 10 grms. of sample, brush into a tared litre flask with cork, add 96 cc. of 5 E HCl and 8 cc. 16 E HNO_3; heat until dissolved, dilute to 840 cc., add 160 cc. of 10 E potassic hydrate solution, saturate with SH_2 (this will take a couple of hours), allow the precipitate to subside, decant as much as possible of the clear supernatant liquor through a filter paper into another weighed litre flask, weigh and calculate for quantity of sample taken as in the example of estimation of Tin (*vide* page 80), remove from flask to a beaker, neutralize with 10 E HCl and add 30 cc. in excess. Allow the precipitate to subside in a warm place for a couple of hours; filter, wash with SH_2 water, and dry in water oven; brush the precipitate of tin, antimony, and arsenic sulphide with the free sulphur produced, into a tared Rose's crucible, provided with a lid and delivery tube; connect the delivery tube with the SH_2 apparatus, and pass current of dry SH_2 over; heat the crucible gently at first with a Bunsen flame, and gradually more strongly, so as to volatilize the free sulphur and arsenic sulphide. The sulphides of Sb and Sn now remain. Moisten with a couple of drops of 16 E HNO_3, add 1 cc. of 22 E HNO_3, allow to digest hot for half-an-hour, evaporate carefully, heat gradually, at first with lid on over Bunsen, and afterwards to red heat with lid off. Cool and weigh the SnO_2 and Sb_2O_4 thus produced, and subtract from this the weight of Sb_2O_4 as found in the other portion taken for the antimony estimation. Remainder = weight of SnO_2. Multiply this by ·7867, divide by amount of sample taken, multiply by 100 = percentage of tin in the sample.

[Test the residue for Cu, which, if present, must be extracted by means of nitric acid, and the residue again ignited and weighed.]

Estimation of Copper (*Brown's Process*).—It sometimes happens that an estimation of the amount of copper present in a sample of copper is sufficient in many cases. But in order to reduce the error involved in its estimation to a minimum, great care is required and perfect confidence and experience in the method on the part of the operator to insure accuracy. The author has found the following modified process of Brown's to give very good results:—Dissolve ·5 grm. of sample (weighed to $\frac{1}{10}$ of a milligram) in 10 cc. of 6 E HNO_3, add 6 cc. 5 E H_2SO_4, evaporate to dryness and heat residue on sand bath until fumes cease to come off; dissolve in 10 cc. of E H_2SO_4, filter off sulphate of lead on a very small filter, wash with E H_2SO_4 until free from copper, dilute to 40 cc., gradually add an E solution of Na_2CO_3 until a slight permanent precipitate is produced in the cold, re-dissolve this in 1 cc. of 5 E H_2SO_4 and add 12 cc. of 2 E sodic acetate solution and 9 cc. of 5 E acetic acid; boil for ten minutes, filter off the basic acetate of iron, &c., without losing a drop of the solution, and wash with a hot $\frac{E}{10}$ solution of sodic acetate till free from copper salt. Reduce filtrate by evaporation to about 35 cc. and wash into a colourless 250 cc. stoppered bottle. Volume now 50 cc.; add to this 50 cc. of a solution of potassic iodide containing 5 grms. KI; shake well, and allow to stand for ten minutes. Now carefully run in from a burette an $\frac{E}{10}$ standard solution of hyposulphite of soda (thiosulphate $Na_2S_2O_3$) until nearly all the free iodine liberated is seen to have disappeared; now add 5 cc. of starch solution (1 grm. starch in 100 cc. of water), which will form blue iodide of starch; run in the hyposulphite very carefully, stirring, until the solution becomes colourless. Note the volume of hyposulphite required. Taking the atomic weight of copper as 63·1, then 1 cc. of $\frac{E}{10}$ $Na_2S_2O_3$ should equal ·00631 grm. copper; but the value of the hyposulphite should invariably be obtained by treating 0·5 grm. of pure electrotype copper as above and noting the volume of the $\frac{E}{10}$ hyposulphite that this is equivalent to. A factor can thus be obtained for determining unknown quantities. Owing to a little difficulty that presents itself sometimes in determining the end of the reaction, due to differences in tint, a duplicate assay should go on at the same time, so that if the first assay was run a little over or a little under the mark, by a second observation this could be corrected. An example will make matters clearer.

·5 grm. of a sample of electrotype copper supplied by Messrs. Hopkin and Williams required 80 cc. of $\frac{E}{10}$ hyposulphite (theoretical quantity being 79·2 cc.) and a sample of tube-plate copper previously completely analyzed by the foregoing methods and giving 99·80 per cent. of copper by difference, required 79·8 cc. $\frac{E}{10}$ hyposulphite. Then

$$80 : 79{\cdot}8 :: {\cdot}5 : x = {\cdot}49875$$

$${\cdot}49877 \times 2 \times 100 = 99{\cdot}75 \text{ per cent. copper present.}$$

Reactions:

$$2\,Cu(NO_3)_2 + 4\,KI = Cu_2I_2 + 4\,KNO_3 + I_2$$

$$2\,Na_2S_2O_3 + I_2 = 2\,NaI + Na_2S_4O_6$$

Determination of Arsenic in Copper (*Pattinson's Process*).—The principle of this process depends upon the fact that if a nitric acid solution of copper be neutralized with caustic soda and a slight excess added, the whole of the arsenic present will be precipitated as arsenate of copper before the precipitation of hydrated cupric oxide, and also that if a neutral solution of nitrate of copper containing arsenic in solution be treated with hydrated oxide of copper the arsenic is likewise precipitated as copper arsenate. Dissolve 10 grams of the copper in 70 cc. of 8E nitric acid, boil off excess of acid, dilute to 250 cc., neutralize with E caustic soda solution, and add a volume of $\frac{E}{10}$ sodic carbonate containing 2½ times as much Na_2CO_3 as the weight of arsenic expected to be present; boil for about 5 minutes, and allow the precipitate to subside and the liquid to become cool; filter—washing of the precipitate is unnecessary—dissolve the precipitate in 20 cc. of hot 5E HCl, dilute to 250 cc., neutralize with 5 E AmHo, and add 40 cc. of 20 E AmHo in excess, saturate with SH_2, and allow to stand at a temperature near the boiling point for 1 hour, filter off sulphide of copper, &c.; the arsenic is now in solution as sulphide; wash with $\frac{E}{10}$ Am_2S solution; dilute filtrate to 230 cc., and carefully add 20 cc. of 10 E HCl, and allow the sulphide of arsenic produced to settle in a warm place for a couple of hours, after which it may be filtered off, washed, dissolved in nitric acid, and the arsenic precipitated with magnesia mixture, and determined as described on page 67.

Distillation Method for Arsenic Estimation.—The principle of this process is that when an arsenical copper is distilled with a solution of ferric chloride in hydrochloric acid, the copper is dissolved as chloride, while the arsenic is distilled as chloride. Weigh out 10 grams of the copper into a 300 cc. retort attached to a Liebig's condenser, and pour on the metal 200 cc. of a solution of ferric and calcic chlorides in hydrochloric acid prepared by dissolving 300 grams of ferric chloride and 600 grams of calcic chloride in 600 cc. of 10 E hydrochloric acid and diluting to the litre with distilled water. Distil until 100 cc. has come over, add 20 cc. of 5 E HCl to the residual liquid in the retort, and continue the distillation until another 50 cc. has come off. The whole of the arsenic is now in the distillate; neutralize the solution with caustic soda, and add 8 grams in excess, saturate with sulphuretted hydrogen, allow to stand in a warm place for some time, filter off any precipitate, and wash with a little $\frac{1}{2}$ E Na_2S solution; dilute filtrate to 230 cc., and carefully add 25 cc. of 10 E HCl; allow the precipitate of sulphide of arsenic to subside in a warm place for a couple of hours, filter and wash, and treat the precipitate and estimate the arsenic therein as described on page 67.

Analytical Data of a Complete Analysis of a Sample of Copper by the foregoing Methods.

Residue insoluble in 6 E HNO_3.

50 grms. taken.

	Grms.
Watch glasses + clip + filter paper after drying in water oven for 2 hours	38·8997
Ditto + insoluble residue dried 1 hour in water oven	38·9097
” ” ” ” ” 2nd time	38·9097
Insoluble residue in 50 grms.	·0100

·01 × 2 = 0·02 per cent. insoluble residue.

Estimation of Iron, Nickel, &c.

Calculation for amount of sample taken.

Original sample taken = 10 grms.

	Grms.
Flask (1) + cork	131·92
Do. + liquid + CuS	1190·08
liquid + CuS	1058·16
CuS (corresponding to 10 grms. Cu)	15·07
Total liquid	1043·09
Flask (2) + cork	128·16
Ditto + filtered liquid	966·02
Liquid taken for analysis	837·86

1043·09 : 837·86 : : 10 : x = 8·032

Equivalent to 8·032 grms. of copper taken for the estimation of iron, nickel, &c.

IRON.

8·032 grms. of sample taken.

	Grms.
Porcelain Crucible marked x	7·4512
Do. + Fe_2O_3 1st ignition	7·4535
" " 2nd ignition	7·4535
Fe_2O_3 . .	0023
Fe_2O_3 into Fe	·7000
Fe . .	·00161

$$\frac{\cdot 00161}{8\cdot 032} = \cdot 00020$$

·0002 × 100 = ·02 per cent. iron.

NICKEL.

8·032 grms. of sample taken.

	Grms.
Porcelain Crucible marked O	7·4514
Do. + NiO 1st ignition	7·4599
" 2nd "	7·4599
NiO	·0085
NiO into Ni . .	·7867
	39335
	62936
	·00668695

$$\frac{\cdot 006687}{8\cdot 032} = \cdot 000832$$

Equivalent to ·083 per cent. nickel.

ZINC.

8·032 grms. of sample taken.

Nil.

MANGANESE.

Nil.

ESTIMATION OF ARSENIC.

10 grms. of sample taken.

	Grms
Porcelain Crucible marked 12	6·0715
Do. + $Mg_2As_2O_7$ 1st ignition	6·2364
" 2nd ignition	6·2360
	·1645

	Grms.
	·1645
Allowing for solubility of precipitate	·0032
	·1677
$Mg_2As_2O_7$ into As . . .	·4839
	15093
	5031
	13416
	6708
	·08115003

08115 × 10 = ·812 per cent. arsenic.

ESTIMATION OF ANTIMONY.

10 grms. of sample taken.

	Grms.
Porcelain Crucible marked *oo*	6·9265
Do. + Sb_2O_4 + F.A.	6·9276
„ „ 2nd ignition	6·9276
Sb_2O_4 . . .	·0011
Sb_2O_4 into Sb . .	·7922
	·7922
	·7922
Sb. .	·00087142

·000871 × 10 = ·009 per cent. antimony.

ESTIMATION OF LEAD.

10 grms. of sample taken.

	Grms.
Porcelain Crucible marked *V*	6·9168
Do. + $PbSO_4$ 1st ignition	6·9286
„ 2nd „	6·9286
$PbSO_4$ . . .	·0118
$PbSO_4$ into Pb .	·6832
	54656
	6832
	6832
	·00806176

·008061 × 10 = ·081 per cent. lead.

ESTIMATION OF BISMUTH.

10 grms. of sample taken.

	Grms.
Porcelain Crucible marked *u*	7·4906
Do. + Bi_2O_3 1st ignition	7·4959
,, 2nd ,,	7·4954
,, 3rd ,,	7·4954
Bi_2O_3	·0048
Bi_2O_3 into Bi	·8965
	71720
	35860
	·00430320

·0043032 × 10 = ·043 per cent. bismuth

ESTIMATION OF SILVER.

10 grms. of sample taken.

	Grms.
Porcelain Crucible marked *x y*	6·0715
Do. + AgCl 1st ignition	6·0724
,, 2nd ,,	6·0724
AgCl	·0009
AgCl = Ag	·7527
	·00067743

·00067743 × 10 = ·007 per cent. of silver.

ESTIMATION OF SULPHUR.

10 grms. of sample taken.

	Grms.
Porcelain Crucible marked *x y*	6·0720
Do. + $BaSO_4$ 1st ignition	6·0761
,, 2nd ,,	6·0761
	·0041
Deduct for sulphur in reagents	·0031
	·0010

·001 × ·1373 = ·0001373

·0001373 × 10 = ·001 per cent. sulphur.

ESTIMATION OF TIN.

Calculation of amount of sample taken.

Original sample taken = 10 grms.

	Grms.
Flask (1) + Cork	132·55
Do. + Liquid + CuS	1201·33
Liquid + CuS . . .	1068·78
CuS	15·07
Total liquid . . .	1053·71
Flask (2) + Cork	128·20
Do. + decanted liquid (filtered)	889·80
Liquid taken for analysis .	761·60

Then 1053·71 : 761·6 : : 10 : x = 7·228

Equivalent to 7·228 grms. sample taken for tin estimation.

	Grms.
Porcelain Crucible marked *T*	7·6287
Do. + SnO_2 + Sb_2O_4	7·6314
,, 2nd ignition	7·6314
	·0027
Deduct Sb_2O_4 (*vide* page 35) $\frac{\cdot0011 \times 7\cdot228}{10}$ =	·0008
SnO_2 . . .	·0019

$$\frac{\cdot0019 \times \cdot7867}{7\cdot228} = \cdot000206 \text{ grm.}$$

Equivalent to ·021 per cent. tin.

ESTIMATION OF OXYGEN.

	Grms.
Watch glass	12·602
Do. + Sample	51·485
Sample taken	38·883
Weight of watch glass + sample after digestion in $AgNO_3$	46·559
Sample dissolved	4·926

The precipitated Ag + $Cu(NO_3)_2 . CuO$, was digested with 5 cc. E $AgNO_3$ and 25 cc. *standard* $\frac{E}{4}$ H_2SO_4, for one hour at 50° C., filtered and washed. On titrating back with $\frac{E}{4}$ Na_2CO_3 until a slight permanent precipitate was obtained on boiling, 23·9 cc. were required.

25 − 23·9 = 1·1 cc. $\frac{E}{4}$ H_2SO_4 used.
1 cc. $\frac{E}{4}$ H_2SO_4 = ·002 grm. O. or ·0178 grm. Cu_2O.
1·1 × ·002 = ·0022 grm. O.
$\frac{·0022}{4·926}$ = ·000446 grm. O.
·000446 × 100 = ·045 per cent. combined oxygen.

1·1 × ·0178 = ·01958 grm. Cu_2O
$\frac{·01958}{4·926}$ = ·004

·004 × 100 = ·40 per cent. suboxide of copper, Cu_2O.

ANALYSES OF TWO SAMPLES OF COPPER.

(By the Author.)

	No. 1. Per cent.	No. 2. Per cent.
Antimony	0·020	0·002
Arsenic	1·010	0·020
Bismuth	0·045	0·021
Iron	0·020	0·005
Lead	0·085	0·023
Nickel	0·083	0·069
Silver	0·010	0·014
Sulphur	Trace	Nil
Tin	„	0·018
Zinc	„	Nil
Oxygen	0·055	0·023
Copper (by difference) . .	98·672	99·805
Total . . .	100·000	100·000

No. 1 is a sample of copper manufactured some forty years ago, and which has done good service as a locomotive fire-box. No. 2 is a sample of recent manufacture: it is a very good sample of copper for electrical purposes, &c., but owing to its purity and consequent softness, it did not last long as a locomotive fire-box.

The specification for copper tube plates for the Indian State Railways enacts that "the plates must be of the very best quality, and samples are to stand a test of being doubled cold without showing any signs of cracking. As many samples of the copper as the Inspector-General of the Rivay Stores shall think fit will be analysed by a metallurgist selected by the Inspector-General. Should any of the analyses show more than 0·5 per cent. in all of other metals or matters, the plates represented by that analysis will be rejected."

The writer's experience with regard to the quality of copper tube plates is that pure copper is too soft to be economically used; but what is the best hardening ingredient to add to copper so as to reduce corrosion and abrasion to a minimum, has yet to be determined; although arsenic in limited quantities would seem a favourable addition for that purpose.

LEAD.

The chief source of lead is from *galena*, which is a sulphide of the metal (PbS), and containing 86½ per cent. of the element when in a pure state. It is found associated with limestone in Derbyshire and Cumberland, and with clay slate in Cumberland. The ore is also abundant in the United States, Spain, and Germany. Other ores of lead occurring in smaller quantities are the *carbonate* (cerusite), *sulphate* (anglesite), and *phosphate* (pyromorphite). Galena always contains a notable quantity of silver, which in smelting the ore is reduced along with the lead, from which it is profitably extracted by a process of concentration, even if the silver is present only to the extent of a couple of ounces per ton of lead. Before smelting, lead ores are generally first concentrated, which is easily performed (owing to their high specific gravity) by mechanical means, by which the greater portion of earthy matter is separated. In obtaining lead from galena it is first roasted in a reverberatory furnace, so as to become partially converted into oxide of lead and sulphate of lead; the unoxidized galena is then mixed up with the oxygenated compounds and melted together, when they re-act upon each other, the whole of the sulphur being eliminated as sulphurous acid gas, while the lead is reduced to the metallic state, after which it is refined, the silver extracted, and cast in pigs for the market.

Pure lead is a soft, white metal, with a bluish white tint. It has a specific gravity of 11·38, and melts at about 330° C. It is very malleable, but not elastic. Owing to the unique properties possessed by lead, its applications are varied and extensive. It is a constituent of many important alloys, such as type-metal, stereo-metal, Britannia metal, pewter, Queen's metal, solder, &c.; it is used for lining vessels for storing, or in the manufacture of the strong acids, such as hydrochloric, sulphuric, hydrofluoric, &c., which have little action upon the metal. Bullets, shots, piping, sinks, are among the many uses lead is put to.

Commercial unrefined lead is much harder than the refined metal, owing to the presence of impurities such as copper, silver, iron, and antimony. A sample of commercial lead examined by the writer gave on analysis :—

	Per cent.
Lead . . .	98·550
Antimony . .	1·102
Copper. . .	0·175
Iron . . .	0·102
Silver . . .	0·022 (= 8 gs. per ton of lead)

which after refining gave :—

	Per cent.
Lead . . .	99·955
Antimony . .	·006
Copper. . .	·025
Iron . . .	·004
Silver . . .	0·001

Method for Analysis of Lead.

Estimation of Silver.—Weigh out 200 grams of the metal in a 500 cc. beaker, and gradually add 6 E nitric acid, in small quantities at a time, until about 180 to 190 grams of the metal has gone into solution, and there is no further action of the acid on it, all the silver is concentrated in the residual metal. The solution of nitrate of lead is poured off, and the remaining metal is dissolved in about 50 cc. 6 E HNO_3 and diluted to 500 cc., and filtered if necessary; to the clear solution add 5 cc. of $\frac{E}{5}$ HCl, warm the solution to about 70° C. for a short time, and allow to stand in a dark place for a day or so. The whole of the silver is now precipitated as chloride (AgCl). The supernatant liquid is now decanted from the precipitate, as far as possible without disturbing the latter, and filtered on to a small ashless paper, and washed free from lead salt with hot distilled water, dried and treated in the same manner as described for the estimation of silver in copper, page 71.

Estimation of Antimony, Copper, and Iron.—Take 100 grams of the sample and treat with 250 cc. of 6 E HNO_3, heat and add small quantities of water so that no nitrate of lead separates, dilute to 500 cc. and allow to stand about twelve hours. If there is any precipitate formed it is filtered off, and reserved to be subsequently tested for antimony. *Residue* 1.

The filtrate is diluted to 1,000 cc., and 30 cc. of 36 E sulphuric acid added, and stirred, the precipitate of sulphate of lead formed is allowed to subside, the supernatant liquid is syphoned off, and the residue washed by decantation with E H_2SO_4. The solutions and washings are transferred to an evaporating basin, and evaporated until fumes of sulphuric acid are copiously evolved; cool the liquid and add 40 cc. of water; a precipitate of sulphate of lead occurs here, generally containing small quantities of antimony. After allowing to stand a short time it is filtered off, and dissolved in 10 cc. of

10 E HCl, diluted to 50 cc. and saturated with sulphuretted hydrogen; the precipitate of lead and antimony sulphides are allowed to subside, filtered off, and the sulphide of antimony dissolved out with 10 cc. 2 E ammonic sulphide; dilute to 40 cc., reserve. *Solution* 1.

The filtrate from the lead sulphate is diluted to 100 cc., heated to about 70° C., saturated with sulphuretted hydrogen and allowed to stand in a warm place for a few hours. The precipitate produced is filtered off and washed with sulphuretted hydrogen water, and digested with hot 2 E potassium sulphide, until all antimony and arsenic sulphides are dissolved out; the residue is washed with sulphide of potassium and the filtrate mixed with Solution 1 acidulated with 10 E HCl and the precipitated sulphides allowed to stand in a warm place for some time.

The residue insoluble in potassium sulphide is digested with 10 cc. hot 5 E HNO_3 until all that is soluble is dissolved, the filter is carefully incinerated, and the ash added to the nitric acid solution; add 5 cc. of 5 E H_2SO_4 and evaporate, until sulphuric acid fumes are evolved; dilute to 20 cc., allow any sulphate of lead to settle, filter with a little E H_2SO_4, dilute to 50 cc., saturate with H_2S, allow sulphide of copper to settle in a warm place, filter, and wash precipitate with H_2S water; reserve filtrate, dissolve residue in 10 cc. of 8 E HNO_3, dilute to 50 cc., heat nearly to boiling, add 2 E sodium hydrate in slight excess, allow the precipitated oxide of copper to subside, filter, and estimate as described on page 94. The filtrate from the sulphide of copper is heated near the boiling point, and a slight excess of 20 E ammonic hydrate is added, boiled, and the hydrate of iron filtered off, washed, and the iron determined as described on page 65.

The precipitated sulphides of antimony and arsenic are now filtered off, treated with fuming nitric acid, separated and estimated as described on page 97. The insoluble residue (Residue 1) obtained on treatment of the original sample with nitric acid is examined, and any antimony obtained added to that found as above.

ZINC.

Zinc is a metal now much used by engineers; it is employed in the form of sheets for various purposes; it protects iron from rusting; it also is used as an electro-positive element in several batteries, and is a constituent of a large number of useful alloys.

The chief ores of zinc are *red zinc ore* (oxide), *calamine* (carbonate), and *zinc blende*, or *black Jack* (sulphide). The ores do not occur largely in this country, most of the zinc used being smelted and imported from Belgium, Silesia, and Poland. The common name for cast zinc is "spelter."

Commercial zinc is a hard, crystalline, and brittle metal, of a white colour with a bluish shade. The pure metal is malleable at the ordinary temperature. Its specific gravity is 6·9 when cast, but which, however, is increased on rolling or hammering to 7·1. It does not contract much on solidification from the liquid condition, and is thus well adapted for castings. Zinc melts at 412° C., and boils at 1040° C.; heated strongly in the air, it takes fire and burns with a greenish-white flame, with the formation of dense fumes of zinc oxide (ZnO).

Methods of Analysis.—Commercial zinc may contain as impurities lead, tin, iron, copper (cadmium). Weigh out 10 grams of the sample and transfer to a 200 cc. beaker, add 60 cc. of 8 E HNO_3, and heat gently until all that is soluble is dissolved, evaporate nearly to dryness, dilute to 100 cc., allow to stand for some time, filter off any oxide of tin, wash with ½ E HNO_3, and estimate tin as described on page 92. The solution is now evaporated to complete dryness, the residue digested with 12 cc. of 10 E HCl, and diluted to 200 cc.; it is then saturated with sulphuretted hydrogen, and any black precipitate of sulphides of lead and copper filtered off and washed quickly with SH_2 water; evaporate solution (1) to 150 cc., and reserve. The black precipitate is treated on the filter with 10 cc. of 8 E HNO_3, and any undissolved sulphur is to be gently incinerated and the ash, which will contain oxide and sulphate of lead, &c., transferred to the solution; 1 cc. of 36 E H_2SO_4

is now added, and the whole evaporated to dryness on the sand bath; moisten the residue with 4 cc. of 5 E H_2SO_4, dilute to 20 cc., allow the precipitate of sulphate of lead to settle, filter and wash with E H_2SO_4, and determine as described on page 93. Should the solution be colourless, there will not be much in the way of copper present; a trace which would not colour the solution would be detected by making the solution alkaline with 20 E AmHo, when a deep blue solution would result. Should there be an appreciable quantity of copper present, it should be precipitated as hydrated oxide with 2 E NaHO solution, and estimated as described on page 93.

The reserved filtrate (1) containing the iron is now made alkaline with 20 E AmHo, and an additional quantity is to be gradually added until all the precipitated hydrate of zinc is completely dissolved; the solution is now boiled, and the hydrate of iron containing some zinc salt is filtered off and washed with E AmCl. Dissolve the precipitate on the paper with 10 cc. of 5 E HCl, wash the iron salt out of the paper with hot distilled water, dilute, filtrate to 50 cc., add 5 cc. of 5 E AmCl, and gradually add 5 E Am_2CO_3 until a *slight* but permanent precipitate is produced; now boil for 5 minutes, and filter off the hydrate of iron which is now free from zinc, and wash with hot water until free from chlorides; dry in water oven, incinerate in a weighed crucible, cool, and weigh. Weight of $Fe_2O_3 \times 70 =$ per cent. iron.

The following are analyses made by the writer of some samples of commercial zinc :—

	(1)	(2)	(3)	(4)
Lead	1·480	1·145	1·320	0·397
Tin	1·020	Trace	Trace	Nil
Copper	0·182	„	„	„
Iron	·217	0·063	0·113	0·020
Zinc (by diff.) . . .	97·101	98·792	98·567	99·583
	100·000	100·000	100·000	100·000

It is most essential in cases where zinc is to be used for electrical purposes that it should be as pure as possible, since small quantities of impurities materially reduce its conductivity. The specification for zinc to be used for electrical purposes should enact that the total impurities must not exceed 0·5 per cent., and of this iron must not exceed 0·04 per cent.

ALLOYS.

Mixed metals or alloys are now largely used in the arts and manufactures. Mixtures of certain metals in definite proportions have many valuable physical properties which any one element of their composition does not possess; thus pure gold is so soft that for coinage and jewellery it is necessary to mix it with a baser metal, such as copper, which confers upon it greater hardness, and therefore greater resistance to abrasion.

Copper, again, in a pure state is too soft to resist the wear necessary for coinage, and hence is mixed with about 4 per cent. of tin and 1 per cent. of zinc, which very materially increases its hardness. If two parts of copper and one part of tin be melted together, a metal is produced which is susceptible of a very fine polish, and is used as a mirror for optical instruments, &c. It is now generally thought that the metals combine with each other in definite chemical proportions, producing compounds which are soluble in any one of its constituents over and above the quantity required to form a definite chemical compound. There are three methods of preparing alloys.

(1.) By fusion—(*a*) mixing the molten metals together, or (*b*) dissolving a solid metal in a molten metal of higher melting point.

(2.) By subjecting the powdered metals to intense pressure.

(3.) By electro-deposition.

The usual method in forming alloys is, of course, by the fusion method.

Of recent years many new alloys, more notably bronzes, have been brought into use for certain purposes; thus *Manganese Bronze* is found to be non-corrosive, and is well adapted for such purposes as ships' propellers, &c. It is of a yellowish-grey colour, and has a tensile strength of about 39 tons per square inch when cold rolled, and has the following percentage composition:—copper, 82·4; manganese, 16·0; iron, 1·5; carbon, 0·1.

Phosphor Bronze, invented by Dr. Kuntzel, is a very hard alloy, and is well adapted for machine parts, bearings, &c. It contains in 100 parts copper, 82; tin, 13; lead, 4·3; phosphorus, 0·7.

Silicon Bronze, copper containing a very small quantity of silicon, is well adapted for telegraph purposes.

Aluminium Bronze is the strongest of the copper alloys. It is composed of copper 90 per cent., and aluminium 10 per cent.; it has a gold-like colour, and when rolled into plates has a tensile strength of 49 tons per square inch.

Delta Metal, an alloy well adapted for bearings. It has a yellow colour, melts at about 1800° F., and has a tensile strength of 40 tons per square inch. It contains in 100 parts copper, 54·5; zinc, 40·4; iron, 1·0; *lead*, 4·0; phosphorus, 0·10.

Muntz's Metal, or malleable brass, is an alloy of 3 parts of copper and 2 parts of zinc; now much used for the sheathing of ships.

Ferro-manganese is a triple alloy of iron, carbon, and manganese, the latter often amounting to over 85 per cent.; used in steel-making.

Spiegeleisen, like ferro-manganese, is a triple alloy, but containing only from 10 to 20 per cent. of manganese.

Methods of Analysis of Alloys.

SPIEGELEISEN AND FERRO-MANGANESE.

Estimation of Manganese.—There are two methods generally in use, (1) Direct method, and (2) Indirect method.

Direct method.—Weigh out ·5 grm. of the finely-powdered sample into a 50 cc. assay flask, add a mixture of 10 cc. 10 E HCl and 5 cc. 22 E HNO_3 (fuming), and boil until all action ceases. Now add a few crystals of potassic chlorate to ensure complete oxidation of organic matter, and boil until all free chlorine is eliminated, and bulk of liquid is reduced to about 8 cc. ; pour into a 1½ litre flask, wash out with water, dilute to 250 cc., and gradually add 5 E ammonic carbonate solution, until a slight permanent precipitate is obtained which does not disappear on vigorously shaking. Dissolve the precipitate in two or three drops of 10 E HCl and add 2 cc. in excess. Now add 20 cc. of 5 E ammonic acetate solution, dilute to 800 cc., boil for ten minutes, filter off quickly the basic acetate of iron thus produced, and wash three times with hot distilled water. The precipitate is dissolved off the paper with 5 cc. 10 E HCl into a 1½ litre flask, diluted to 250 cc., and iron precipitated and filtered off as before. The two filtrates are now mixed in a large beaker, and evaporated down to 1 litre, allowed to cool, 3 cc. of bromine added, and stirred until all goes into solution. The rest of the process is conducted as stated on page 43.

Indirect method.--Estimate the percentage of iron volumetrically by the process described on page 53, add to this result 6 per cent. for carbon and impurities, and the difference between this and 100 is taken as the percentage of manganese present. This indirect method is largely used in Spiegel works, &c.; and where a number of estimations are required in one day, it may be considered sufficiently accurate for all practical purposes. In cases of dispute between buyers and sellers the direct method must be adopted. Fresenius gives the following analysis of Spiegeleisen made from Stahberg spathic ore.

	Per cent.
Iron	82·860
Manganese	10·707
Nickel	0·016
Cobalt	trace
Copper	0·066
Aluminium	0·077
Titanium	0·006
Magnesium	0·045
Carried over	93·777

	Per cent.
Brought forward	93·777
Calcium	0·091
Potassium	0·063
Arsenic	0·007
Antimony	0·004
Phosphorus	0·059
Sulphur	0·014
Nitrogen	0·014
Silicon	0·997
Carbon	4·323
Slag	0·665
	100·014

Manganese can be made to replace iron in pig iron to the extent of 85 per cent. When pig iron contains manganese from about 5 to 25 per cent. it is called spiegeleisen, and when over this amount ferro-manganese. Manganese plays an important part in the manufacture of steel. Owing to its greater affinity for oxygen than iron it deoxidizes any FeO formed in the process; the resulting MnO forming a fluid slag with the impurities and readily separating from the molten metal. It likewise neutralizes to a great extent the deleterious effects of sulphur.

FERRO-ALUMINIUM.

Estimation of Aluminium.—Weigh out 5 grams of the sample in a 500 cc. beaker, and dissolve in 70 cc. of 10 E H_2SO_4, evaporate to dryness, redissolve residue in 60 cc. of 2 E H_2SO_4, dilute in a graduated measure to exactly 300 cc., and well mix; 100 cc. of the solution (= 1·666 grams) are filtered off into a graduated 100 cc. measure; this is then poured into a 250 cc. beaker; about 5 grams of pure iron wire is now added and the solution boiled, so as to reduce any persalt of iron formed; the excess of acid is carefully neutralized with 2 E $NaCO_3$, and the mixture gradually poured into 150 cc. of a boiling solution containing 30 grams of potassic hydrate and 60 grams of potassic cyanide; the mixture of potassic hydrate and potassic cyanide with iron precipitated as hydrate is diluted up to 500 cc. in a graduated measure, and 300 cc. (= 1 gram of sample) filtered off into a large evaporating dish; 200 cc. of an E solution of ammonium nitrate are now added and the mixture heated for about an hour; filter and wash the precipitated alumina with hot water, redissolve in 20 cc. of 5 E HCl, dilute to 200 cc., neutralize with 20 E AmHo, and add 10 cc. in excess, boil, filter and wash with hot water, dry and ignite and weigh as Al_2O_3. The weight obtained multiplied by 0·534 × 100 = percentage of aluminium. Aluminium is generally present to the extent of 12½ per cent.

BRASS, BRONZE, &c.

Estimation of Tin, Lead, Copper, Iron and Zinc.—The following process is applicable to the analysis of Aich's metal, bell metal, brass (all colours), bronze for bearings, Dutch metal, gun metal, Muntz's metal and sterro metal. Although all the above-named elements are not always present in these alloys, it is always advisable to pass them through the process. If it be thought that the amount of any one element present, judging from the amount of precipitate found, is not sufficient in quantity to affect the quality of the alloy, of course it can be filtered off and discarded, and the estimation of the more important metals pursued.

Proceed by weighing out 1 grm. of the sample into an 80 cc. low and wide beaker, add 15 cc. of 6 E HNO_3, and heat until all that is soluble is dissolved. If any quantity of tin be present a white precipitate of an hydrate of tin, called metastannic acid, is produced. Evaporate on the water bath nearly to dryness, dilute with distilled water to 100 cc., boil, filter off the precipitate, wash with $\frac{1}{10}$ E HNO_3, until the filtrate gives no reaction for copper with potassic ferrocyanide, and then with distilled water until free from acid. The filter and its contents are spread out on a watch glass and put into a water oven to dry; in the meantime a small porcelain crucible is weighed, after which the precipitate is brushed into it, the filter ignited separately and the ash added to main portion, finally ignited for ten minutes over the blow-lamp, and weighed. The weight of the oxide of tin (SnO_2), thus produced × ·7867 × 100 = percentage of tin present. The filtrate, which may now contain lead, copper, iron and zinc as nitrates, is evaporated as low as possible on the water bath, with addition of 2 cc. of 36 E H_2SO_4, and finally on the sand bath until nearly all fumes of H_2SO_4 are driven off. Digest residue with 50 cc. $\frac{E}{2}$ H_2SO_4 till all that is soluble is dissolved, filter off $PbSO_4$, wash with $\frac{E}{2}$ H_2SO_4 until free from copper, and finally with water until free from acid. Dry in water oven, scrape precipitate into weighed porcelain crucible, ignite filter separately, add ash to main portion, ignite at a dull red-heat for ten minutes, and weigh. Weight of $PbSO_4$ × ·6832 × 100 = percentage of lead. The filtrate is mixed with 23 cc. 10 E HCl, and diluted to 350 cc., heated to 70° C., saturated with sulphuretted hydrogen, and the precipitated copper sulphide allowed to subside, and then filtered off and washed quickly with SH_2 water.

To ensure the perfect separation of zinc, the precipitate is dried, roasted carefully in a porcelain capsule, dissolved in 10 cc. of aqua regia (3 cc. 16 E HNO_3 and 7 cc. 10 E HCl), evaporated to dryness, residue dissolved in 52 cc. of 5 E HCl, and diluted to 350 cc., and the copper separated as sulphide as before; the two filtrates are mixed and reserved for the determination of zinc and iron. The CuS is dissolved off the filter with 10 cc. of 8 E HNO_3, using small portions at a time, and collected in a 300 cc. porcelain basin; the filter is washed free from Cu, the solution diluted to 200 cc., and heated nearly to boiling; 2 E NaHO solution is now run in, until no further precipitation of copper hydrate takes place, and the solution shows a slight alkaline reaction. It is now boiled, allowed to settle, and the clear liquid decanted as far as possible through a filter paper; 150 cc. of hot water are poured on to the residue and again boiled, the whole filtered off, and the precipitate washed with hot water till free from alkali. The filter and its contents are now dried in water oven, the CuO scraped into a weighed crucible, the filter paper ignited separately, the ash added to main portion, and the whole ignited for ten minutes at a dull red heat over a Bunsen. The weight of the CuO thus obtained × ·7985 × 100 = percentage of copper.

The reserved filtrates containing any iron and zinc are evaporated to 200 cc., with addition of a few crystals of $KClO_3$ for oxidation, poured into a 300 cc. porcelain dish, heated nearly to boiling, and 2 E Na_2CO_3 run in gradually, stirring until all the iron and zinc present are precipitated as basic carbonates, and the solution shows a distinct alkaline reaction. It is now boiled, allowed to settle, the clear liquid filtered, the residue washed three times with boiling water by decantation combined with filtration, the precipitate finally washed on the paper until free from alkali, dried in the water oven, scraped into a weighed platinum crucible, the filter paper ignited in usual manner, and ash added to main portion. The crucible and its contents are now heated to bright redness for ten minutes, cooled, and weighed. The increase in weight = $ZnO + Fe_2O_3$. Dissolve the mixed oxides in about 5 cc. of 10 E HCl, wash out into a 250 cc. bulb flask, reduce with 2 E Na_2SO_3, and estimate the amount of $\frac{E}{10}$ $K_2Cr_2O_7$ taken up by the reduced iron on titration by the process given on page 53. The number of cc.'s required × ·0056 × 100 = percentage of iron present in the sample. To ascertain the percentage of zinc let

$$ZnO + Fe_2O_3 = X.$$
$$Fe_2O_3 = Y.$$
$$ZnO = Z.$$
$$Y = \text{cc. } \frac{E}{10}\ K_2Cr_2O_7 \text{ required} \times \cdot008.$$
$$Z = X - Y$$

and $$Z \times \cdot8026 \times 100 = \text{percentage of zinc.}$$

The following is an example of analytical data of a bad sample of brass, by the foregoing process :—

ESTIMATION OF LEAD.

1 grm. of sample taken.

Porcelain Crucible marked W	7·7159
Do. + $PbSO_4$	7·7336
,, ,, 2nd ignition	7·7336
$PbSO_4$	·0177

·0177 × ·6832 = ·01209264.

·01209264 × 100 = 1·21 per cent. Pb.

ESTIMATION OF COPPER.

Porcelain Crucible marked V	8·2226
Do. + CuO + F.Ash.	8·8626
,, ,, ,, 2nd ignition	8·8623
,, ,, ,, 3rd ,,	8·8623
	·6397
F.A.	·0029
	·6368

·6368 × ·7985 × 100 = 50·85 per cent. Cu.

ESTIMATION OF ZINC.

Small Platinum Crucible	13·8316
Do. + ZnO + Fe_2O_3 + F.Ash	14·4401
,, ,, ,, ,, 2nd ignition	14·4385
,, ,, ,, ,, 3rd ,,	14·4385
	·6069
F.A.	·0029
(X) ZnO + Fe_2O_3	·6040
(Y) Fe_2O_3 (see below)	·0496
(Z) ZnO	·5544

·5544 × ·8026 × 100 = 44·49 per cent. Zn.

ESTIMATION OF IRON.

The ·6042 grm. ZnO + Fe_2O_3 was dissolved in 10 E HCl, and the Fe estimated therein by standard $\frac{E}{10}$ $K_2Cr_2O_7$ in the usual manner, when 6·2 cc. were required for complete oxidation.

$$1 \text{ cc. of } \frac{E}{10} K_2Cr_2O_7 = \cdot 0056 \text{ grm. Fe.}$$

$$1 \text{ cc. of } \frac{E}{10} K_2Cr_2O_7 = \cdot 008 \text{ ,, } Fe_2O_3$$

$$6 \cdot 2 \times \cdot 008 = \cdot 0496 \text{ grm. } Fe_2O_3.$$

$$6 \cdot 2 \times \cdot 0056 \times 100 = 3 \cdot 47 \text{ per cent. Fe.}$$

SUMMARY.

	Per cent.
Lead	1·21
Iron	3·47
Copper	50·84
Zinc	44·49
Tin	traces
	100·01

When a sample of an alloy is submitted for analysis, and its nature unknown, it would perhaps be wise to first make a careful qualitative analysis, which may in some cases modify the *modus operandi* of the quantitative analysis.

The following table gives analyses of various alloys, which can be analysed by the process just described.

	Tin.	Copper.	Iron.	Zinc.
Aich's Metal . . .	—	60	1·8	38·2
Bell Metal . . .	22	78	—	—
Brass, B . . .	—	71·4	—	28·6
,, Common . . .	—	66·6	—	33·4
,, Yellow . . .	—	60	—	40
,, Pin Wire . . .	—	63	—	37
Bronze Coinage . . .	4	95	—	1
,, for Bearings . .	16	82	—	2
,, ,, ,, . .	17	6	—	77
,, ,, Wheel Boxes .	18	80	—	2
Dutch Metal . . .	—	84·6	—	15·4
Sterro Metal . . .	1·5	58	3·0	37·5
Speculum Metal . . .	33·4	66·6	—	—
Muntz Metal . . .	—	60	—	40
Stopcocks and Pump Valves	10	88	—	2

WHITE METALS (*method for analysis*).

The following alloys, which may contain tin, antimony, lead, copper, iron or aluminium, such as Britannia metal, Babbit's metal, solder, type metal, and pewter (triple and ley), may be analysed by the process here described. Weigh out 1 grm. of sample in fine shavings or drillings into an 80 cc. low and wide beaker, add 14 cc. 8 E HNO_3, heat nearly to boiling on a hot plate until all that is soluble is dissolved, evaporate nearly to dryness on water bath, add 50 cc. of distilled water to the residue, boil until all that is soluble is dissolved, allow to settle, pour off the clear liquid through a filter, add 20 cc. hot water to the residue, boil, and filter off as before. Continue this treatment three times, and finally filter off the precipitate which may be a mixture of metastannic acid and antimonic acid, and wash with hot water until the washings scarcely redden litmus paper; reserve the filtrate (No. 1), dry the precipitate in the water oven, brush into a silver crucible of about 30 cc. capacity, ignite filter paper, and add ash. Now add to the precipitate 8 times its bulk of pure sodic hydrate (note the weight added), fuse over a Bunsen for a quarter of an hour, treat the fused mass with water containing 33 per cent. by volume of alcohol until all that is soluble is dissolved, transfer to a beaker and dilute with the alcohol solution until the strength of the solution becomes E with the quantity of sodic hydrate taken. It is now allowed to stand with frequent stirring for twelve hours. The stannate of soda and excess of sodic hydrate are now in solution, while the antimoniate of soda is precipitated. Filter off and wash with alcohol (30 per cent.), and finally with strong alcohol. Reserve filtrate (No. 2), dissolve the precipitate off the filter with 25 cc. 2 E HCl, in which there are previously dissolved 4 grms. of tartaric acid, and collect in small flask. Dilute to 100 cc., fit cork into flask into which are fitted two tubes, one bent at right angles and going to the bottom of the flask, and the other bent twice at right angles, and just passing through the cork. Connect with SH_2 apparatus and dip the off-tube bent at right angles into a small beaker of distilled water. Fig. 30 speaks for itself with regard to the fittings.

Pass SH_2 until saturated, applying heat gently at first and finally to the boil. After complete precipitation has been insured, allow the flask to stand on a warm plate for half an hour and then pass CO_2 through until the odour of SH_2 is eliminated. Filter quickly, wash with SH_2 water, dry in water oven, scrape off the antimonious sulphide into a small beaker, ignite the filter, and add ash. Moisten with 16 E HNO_3, gradually add 5 cc. of 22 E HNO_3, heat on plate until oxidation is complete, pour into a weighed porcelain crucible, rinsing out the last portions of precipitate with distilled water by the aid of a wash bottle, evaporate to dryness, and gradually ignite to full red heat. Cool and weigh the Sb_2O_4 thus produced, which × ·7922 × 100 = percentage of antimony.

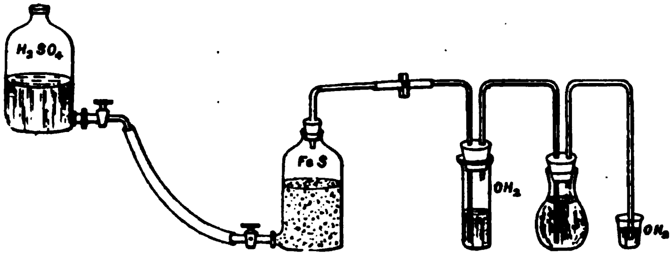

FIG. 30.

The filtrate (No. 2) containing the tin is evaporated until all alcohol is expelled. Neutralize with 10 E HCl, add 5 cc. in excess, dilute to 100 cc., saturate with SH_2, allow to stand in a warm place until SH_2 is nearly all driven off, filter, and wash with a solution containing 5 per cent. 5 E ammonic acetate and 5 per cent. 5 E acetic acid, till free from chlorides. Dry in water oven, scrape precipitate into a watch glass, ignite filter in a weighed crucible, oxidize ash with a drop of 22 E HNO_3, and ignite. Now add main portion from watch glass, place lid on crucible, heat gently at first, then take lid off and increase the temperature gradually until SO_2 comes off freely; when this abates, heat strongly over the blow-lamp for ten minutes, cool, add a small lump of ammonic carbonate, and ignite first over Bunsen with lid on till Am_2CO_3 has all gone off, and finally over the blow-lamp as before. Repeat until no further loss occurs. Cool and weigh. The weight of the SnO_2 thus produced × ·7867 × 100 = percentage of tin present.

To the filtrate (No. 1) containing the lead, copper, &c., add 10 cc. of 10 E H_2SO_4, evaporate to dryness, heat up on sand-bath till all H_2SO_4 fumes have gone off, digest with 30 cc. of E H_2SO_4 until all that is soluble is dissolved; filter off $PbSO_4$, wash with E H_2SO_4 till free from any copper, remove filtrate, wash the acid out of filter with strong alcohol, dry, and proceed as directed on page 93. Evaporate the filtrate to dryness, dissolve residue in 50 cc. E HCl, saturate with SH_2, digest, filter off CuS, washing quickly with SH_2 water, treat and estimate as directed on page 93. The solution, which may contain small quantities of iron and aluminium, is boiled to expel SH_2, and evaporated to about 30 cc., with addition of a few crystals of $KClO_3$ to convert $FeCl_2$ into Fe_2Cl_6. The solution is now made slightly alkaline with 20 E AmHO, 5 cc. 5 E AmCl added, and boiled. If any appreciable quantity of ferric or aluminic hydrate is here precipitated, it is to be filtered off, and the iron and aluminium determined therein as directed on page 111. SH_2 may be passed through the ammoniacal filtrate, and any Zn, Ni, Mn, or Co will be precipitated if present, and may be determined by the methods given in the analysis of copper on page 64.

The following is an example of analytical data obtained in the analysis of a sample of Babbit's metal by the above process:—

ESTIMATION OF TIN.

1 grm. of sample taken.

Porcelain Crucible marked L.	7·1427
Do. + SnO_2 + F.A.	7·9273
" " " 2nd ignition	7·9274
	·7847
F.A. .	·0029
	·7818

·7818 × ·7867 × 100 = 61·50 per cent. tin.

Estimation of Antimony.

Porcelain crucible marked C	7·0916
Do. + Sb_2O_4 + F.A.	7·2267
,, ,, ,, 2nd ignition	7·2264
	·1348
F.A.	·0017
	·1331

·1331 × ·7922 × 100 = 10·54 per cent. antimony.

Estimation of Lead.

Porcelain crucible marked Y	7·8115
Do. + $PbSO_4$ + F.A.	8·2005
,, ,, ,, 2nd ignition	8·2003
$PbSO_4$ + F.A.	·3888
F.A.	·0010
	·3878

·3878 × ·6832 × 100 = 26·49 per cent. lead.

Estimation of Copper.

Porcelain crucible marked F	6·7891
Do. + CuO + F.A.	6·8082
,, ,, ,, 2nd ignition	6·8082
Cu + F.A.	·0191
F.A.	·0010
CuO	·0181

·0181 × ·7985 × 100 = 1·45 per cent. copper.

Summary.

	Per cent.
Tin	61·50
Lead	26·49
Antimony	10·54
Copper	1·45
Iron, Zinc, Aluminium, &c.	traces
	99·98

ANALYSES OF A FEW WHITE ALLOYS.

	Antimony.	Copper.	Tin.	Lead.	Zinc.
Britannia Metal .	6·2	1·8	92	—	—
Pewter, Triple .	15·0	—	79	6	—
,, Ley .	—	—	80	20	—
Solder, Fine .	—	—	66·6	33·4	—
,, Common	—	—	50	50	—
,, Coarse .	—	—	33·4	66·6	—
,, Brazing .	—	50	—	—	50
Type Metal . .	25	—	—	75	—
,, ,, . .	25	—	25	50	—

TIN PLATE.

Estimation of Tin.—It sometimes happens, that a knowledge of the thickness of tin on a tin plate is required. The following method has been proved to give good results:—Weigh out 5 grms. of the sample in chips into an 80 cc. bulb flask provided with a cork, into which passes a tube with a Bunsen valve attached, add 53 cc. of 5 E HCl, dissolve (the object of dissolving the sample out of contact with the air, is to prevent precipitation of a large amount of sulphur by subsequent treatment with SH_2, if the iron were present as perchloride), pour into a 300 cc. beaker, dilute to 250 cc., add 13 cc. of 10 E HCl, saturate with SH_2, allow to stand in warm place for half an hour, filter off SnS, and wash with SH_2 water. Dissolve the SnS off the paper with 12 cc. of hot 2 E Na_2S (in which 0·1 grm. of sulphur is previously dissolved), by frequent percolation, and repeat with 12 cc. more. The solutions are now mixed, diluted to 100 cc., neutralized with 10 E HCl, adding 5 cc. in excess, allowed to stand in warm place for an hour, and the stannic sulphide filtered off and treated as described on page 72, in order to separate any arsenic sulphide that may be present, when the tin may be determined as oxide. A sample of tin plate gave by this process 2·5 per cent. of tin.

WHITE LEAD.

Pure white lead is supposed to be a mixture of carbonate and hydrate of lead, but often it is mixed with, and sometimes wholly substituted by, such substances as chalk, *heavy spar* (barium sulphate), gypsum, *witherite* (baric carbonate), zinc oxide, kaolin, &c. For paints it is usually previously mixed with oil and "dryers," a compound which may contain a great number of substances, often of doubtful efficiency.

Before making a quantitative analysis of a sample, it is well to first subject it to a careful qualitative analysis, and if not pure, to determine the nature and quantity of the adulterants.

Qualitative Analysis.—Weigh out about a grm. of the sample, add 20 cc. of 8 E HNO_3, heat on hot plate until all that is soluble is dissolved, suck up any oil floating on the surface of the liquid with a piece of blotting or filter paper, dilute to about 40 cc., filter, and wash with $\frac{E}{10}$ HNO_3. If any residue is present it may consist of $BaSO_4$, $PbSO_4$, SiO_2, &c.; pour on about 10 cc. of hot 5 E ammonic acetate, pouring it through several times. Any $PbSO_4$ is thus dissolved out, which will give a yellow precipitate of $PbCrO_4$, when a solution of E K_2CrO_4 is added to it. The residue is fused with four times its weight of potassic carbonate and tested in the usual manner for acids and bases. The original nitric acid solution is diluted to about 150 cc., and conducted through the usual groups of reagents. If there is no insoluble residue on treatment with 8 E HNO_3, and if, after the lead has been separated by SH_2 a little of the solution leaves no residue on evaporation on platinum foil, it may be taken for granted that there would not be much present in the way of the usual adulterants.

Method for Quantitative Analysis.—We will assume that the qualitative analysis shows the following to be present:—Oil, $BaSO_4$, $BaCO_3$, $CaCO_3$, $CaSO_4$, $PbCO_3$ and $Pb(HO)_2$. Weigh out 2 grms. of the sample into a two-ounce stoppered bottle, pour on 20 cc. of methylated ether, tie down stopper, heat the bottle and its contents to about 40° C., thoroughly shake, and allow the sediment to subside. When cool, withdraw stopper and decant the clear ethereal solution of the oil through a double filter into a weighed flask. Repeat the digestion with ether three times, finally filtering off the residue, and washing with ether until free from oil.

Distil off the ether by putting the flask, attached to a Liebig's condenser, into a beaker of hot water, and weigh the residual oil. The residue on the filter is dried and brushed into a flask, and the CO_2 determined therein by the method described on page 110, using 8 E HNO_3 for the decomposition instead of HCl. The solution from this determination is now filtered, and the residue, which will consist of $BaSO_4$ and $CaSO_4$, is washed with $\frac{E}{10}$ HNO_3. Reserve solution. The residue is dried, brushed into an 80 cc. porcelain basin, and 20 cc. of 2 E Na_2CO_3 poured on to it and boiled for about an hour, keeping up the original bulk with hot water; the $CaSO_4$ is thus completely decomposed, insoluble calcic carbonate being formed, together with soluble sulphate of soda. The solution is diluted to 50 cc., and the $CaCO_3$ and $BaSO_4$ filtered off. Since the $CaCO_3$ on the filter is the equivalent of the $CaSO_4$ originally present, the solution may be rejected, the $CaSO_4$ being deduced from the amount of CaO found hereafter. Percolate 20 cc. of hot E HCl through the precipitate several times, wash the $BaSO_4$ remaining on the filter, dry, determine in usual manner. Test residue for $PbSO_4$. Dilute the solution to about 30 cc., add 5 cc. 5 E AmCl, 5 cc. of 20 E AmHO, and 10 cc. of E ammonic oxalate; stir well, and allow the calcic oxalate to settle for a few hours in a warm place, after which it is to be filtered off, washed, dried, ignited gradually to a bright red heat, cooled, and the resulting CaO weighed. Every 7 parts of CaO equal 17 parts of $CaSO_4$.

The reserved nitric acid solution containing lead, &c., is now evaporated nearly to dryness, 2 cc. of 10 E HCl added, the solution diluted to 150 cc. with hot water, saturated with SH_2, the PbS allowed to subside, filtered off, washed with SH_2 water, dissolved off the filter in 8 E HNO_3 and precipitated and determined as sulphate as directed on page 93. The filtrate is made alkaline with 10 cc. of 20 E AmHO, 5 cc. of 5 E Am_2S added, and then allowed to stand a short time in a warm place. Any precipitate that may be formed is filtered off and examined for zinc, iron, &c. The filtrate contains the barium and calcium originally present in the sample, as carbonates. Add to the solution 10 cc. of 5 E Am_2CO_3, allow to stand some time, and filter off the carbonate of Ca and Ba through a weighed filter. The solution might be tested for magnesia by adding to it a solution of sodic phosphate, and allowing to stand twelve hours.

It now being necessary to determine the proportion of $CaCO_3$ and $BaCO_3$ present in the precipitate, this may be done indirectly, since there is such a wide difference in the atomic weights of Ba and Ca. Dry the precipitate and weigh. Determine the CO_2 present by the method employed for the estimation of total CO_2, the difference between these two amounts being the quantity combined as carbonate of lead. To calculate the amounts of $BaCO_3$ and $CaCO_3$ from the data obtained—

$$\begin{aligned} \text{Let } X &= \text{weight of } BaCO_3 + CaCO_3\,; \\ Y &= CO_2 \text{ found}\,; \\ Z &= CaCO_3 \text{ in mixture.} \end{aligned}$$

then

$$\begin{array}{lll} CO_2 & EqBaCO_3 & \\ 44 \ : & 197{\cdot}2 & :: Y : C \ (BaCO_3\text{, if all present as such).} \end{array}$$

$$C - X = D \text{ which is proportional to amount of } CaCO_3 \text{ present.}$$

$$\begin{array}{lll} BaCO_3 - CaCO_3 & Ca.CO_3 & \\ 97{\cdot}2 \quad : & 100 & :: D : = Z \ (CaCO_3) \end{array}$$

$$\text{and } X - Z = BaCO_3.$$

PART II.

ORES, LIMESTONES, ETC.

IRON ORES.

Method for Complete Analysis.—The ore, having been carefully sampled, is quartered and powdered moderately fine, taking care that the last portion completely passes through the smallest sieve. It is thoroughly mixed and placed in a dry stoppered bottle.

Estimation of Moisture in Ores which contain no CO_2 or Organic Matter.—Weigh out 2 grms. of the sample in a small shallow dish, and place in a water oven. After being at 100° C. for two hours, it is placed in a desiccator, cooled and weighed, and the loss noted; it is again placed in water oven for half an hour and weighed. If no further loss occurs, all moisture is eliminated. Loss × 50 = percentage hygroscopic water.

Estimation of Water (free and combined) in Ores containing CO_2 and Organic Matter.—Weigh out 3 grms. of the sample into a bulb tube, Fig. 31, A, and attach to a weighed $CaCl_2$ tube, C. This is connected with the cylinder, D, containing 36 E H_2SO_4, which serves to ascertain the rate at which the air comes through, and to absorb any moisture that may be given off from E. E is an aspirator, and B is a tube containing $CaCl_2$, which dries the air previous to its passing through A. A gentle heat is at first applied to the bulb, and air made to aspirate through gently, and the heat gradually increased to redness. After it is assured that all moisture is deposited in the $CaCl_2$ tube C, it is detached and carefully weighed; the increase in weight is the total water free and combined in 3 grms. sample. On subtracting the amount of free moisture obtained as above, the amount of combined water is obtained.

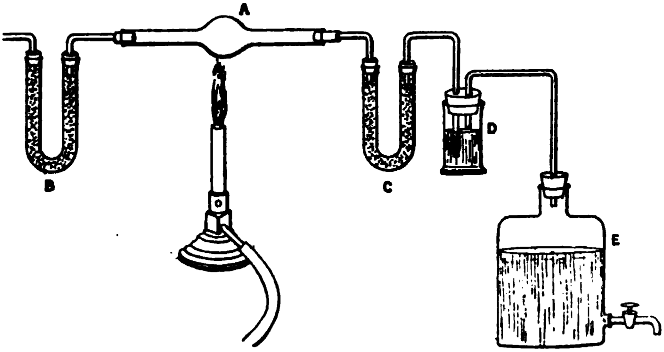

FIG. 31.

Estimation of Total Iron.—Weigh out 1 grm. of the sample into an 80 cc. beaker, add 20 cc. of 10 E HCl, and boil gently for about twenty minutes, or until no further action takes place; some ores are completely decomposable by 10 E HCl, leaving a white insoluble residue, while others are very difficult to act upon. In any case, evaporate to complete dryness and heat on a sand bath to about 130° C., for half an hour, to render silica insoluble; cool, and boil with 10 cc. of 10 E HCl until all that is soluble is dissolved; dilute to 50 cc., filter, and wash insoluble residue with hot E HCl until free from Fe_2Cl_6 (testing washings with KCyS). Reserve filtrate, dry the residue in water oven, ignite at a full red heat in a weighed platinum crucible, cool, and weigh. Increase in weight × 100 = percentage of "Insoluble Residue." Now this insoluble residue invariably contains more or less iron, alumina, &c., and in order to estimate these add four times its weight of Fresenius' flux ($Na_2CO_3 + K_2CO_3$), fuse for twenty minutes over the blow lamp, cool, treat repeatedly with small quantities of boiling water, allow to settle, filter the clear liquid, transfer the bases to the filter with the aid of wash-bottle and feather, and wash till free from alkali. Reserve the filtrate for estimation of silica. The bases are now dissolved off the paper with 5 cc. of 8 E HCl, the paper washed till free from Fe_2Cl_6, and the solution added to that containing the main portion of the iron. To the mixed solutions are now added a few crystals of $KClO_3$ (to ensure perfect peroxidation). Boil down to 50 cc. Transfer to a 250 cc. bulb flask provided with a Bunsen valve, reduce with 2 E Na_2SO_3, and determine the iron volumetrically, as directed on page 49. This gives the total iron, *i.e.* that existing as peroxide and protoxide.

Determination of the Silica.—The solution reserved from the iron estimation for the determination of silica, is rendered neutral with 10 E HCl, 10 cc. added in excess, evaporated to dryness on the water bath, and then heated on the sand bath at about 130° C., to render silica insoluble; 10 cc. of 5 E HCl are now added, then boiled for about ten minutes, diluted to 50 cc., allowed to settle in a warm place, the clear liquor decanted through a filter paper, the residue boiled up with 50 cc. of water, and the whole filtered. The silica, which should now be colourless, is washed till free from chlorides, with boiling water (test washings with $AgNO_3$), dried in water oven, transferred to weighed crucible, ignited at bright red heat, cooled, and weighed. Increase in weight × 100 = percentage of silica.

[The purity of the silica must be tested by evaporating with H_2SO_4 and hydrofluoric acid, and igniting. There should be no residue left; if there is, subtract. This residue will be examined in another portion of the analysis.]

Determination of the Iron existing as Ferrous Oxide (FeO).—Many iron ores contain iron in two degrees of oxidation: the sesquioxide (Fe_2O_3) and the protoxide (FeO). The protoxide may exist in a state soluble and insoluble in HCl. To determine the FeO soluble in HCl, select a 100 cc. bulb flask, into which a rubber cork containing a tube fitted with a Bunsen valve is made to fit, weigh into it about 1 grm. of bicarbonate of soda, and then brush in 1 grm. of sample accurately weighed. Now add 12 cc. of 10 E HCl, and quickly fit in cork; the CO_2 given off by the decomposition of the bicarbonate thus drives the air out of the flask. Apply a gentle heat until the acid is in a state of incipient ebullition, which is continued until no further action is observable; dilute the solution to 200 cc., pour into a 250 cc. beaker, add 10 cc. of 10 E HCl, and titrate the solution with *standard* $\frac{E}{10}$ $K_2Cr_2O_7$, in usual manner. The number of cc.'s required × ·0056 × 100 = percentage Fe existing as soluble FeO, and cc.'s required × ·0072 × 100 = percentage soluble FeO. The FeO existing in the insoluble portion is determined as follows: The insoluble residue in the above solution is filtered off through a plug of asbestos felt, free from iron, and washed with hot water; it is dried and transferred with the felt to a platinum crucible of about 25 cc. capacity. It now becomes necessary to decompose the residue so as to render the insoluble FeO soluble in HCl without undergoing peroxidation; to adopt this, the process recommended by Avery* is suitable. The crucible and its contents are placed on a platinum triangle, fixed over a hole in a special bath, Fig. 32, and 10 cc. of 10 E HCl, and 5 cc. of hydrofluoric acid (HF), poured on. It will be observed that there is a groove, G, in the top of the bath; this is partially filled with water; and the funnel, F, is made to fit into it, thus forming an air-tight joint. A Bunsen flame is put under the bath (which is half filled with water); meanwhile a current of coal-gas is made to pass through the tube, *t*, and the heat continued until the residue and felt are completely dissolved; after which it is transferred quickly to a beaker, diluted to 200 cc., 10 cc. of 10 E HCl added, and the iron present in the ferrous state, titrated by the *standard* $K_2Cr_2O_7$, as before.

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* *Chemical News*, xix., 270.

To ascertain the amount of peroxide of iron (Fe_2O_3) present in the sample, subtract from the amount of standard $K_2Cr_2O_7$ required in the determination of total iron, the amount required for oxidation of ferrous iron in soluble and insoluble portion. This × ·008 × 100 = the percentage of Fe_2O_3 present.

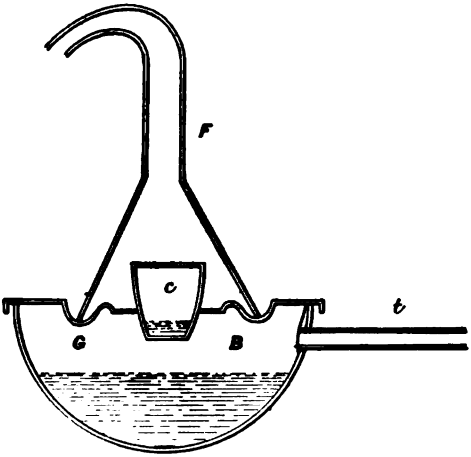

FIG. 32.

Estimation of Carbonic Acid.—Some ores of iron contain a rather large percentage of combined CO_2, such as the spathic ore, blackband ore, &c. If a preliminary test for CO_2 (made by warming some of the ore with 8 E HCl in a test-tube and observing if there is any effervescence) gives a negative result, of course the quantitative estimation need not be proceeded with. The following is a good method for its determination when present. Fig. 33 is a sketch of the apparatus employed. Weigh out 3 grms. of the sample into the flask, D, and connect in the manner as shown below; A is a U-tube containing pieces of caustic soda, which serve to absorb CO_2 of the air that is made to pass through the apparatus by means of the aspirator, I; C is a bulb tube containing 30 cc. of 8 E HCl, which is prevented from flowing out by means of a clip at B; E are bulbs containing a little 36 E H_2SO_4, which serves to absorb the great bulk of moisture coming from D; F is a U-tube containing in the first limb pieces of pumice-stone, which have been soaked in a concentrated solution of copper sulphate, dried, and ignited; this serves to absorb any HCl that comes over; the second limb contains pieces of calcium chloride; G is a weighed U-tube containing in the first three quarters, small pieces of caustic potash; and the remaining quarter, pieces of $CaCl_2$. This tube absorbs the CO_2 liberated, which is to be weighed before and after experiment; H is a tube containing pieces of $CaCl_2$, which serve to absorb any moisture from air that may tend to come into G from I. When all is

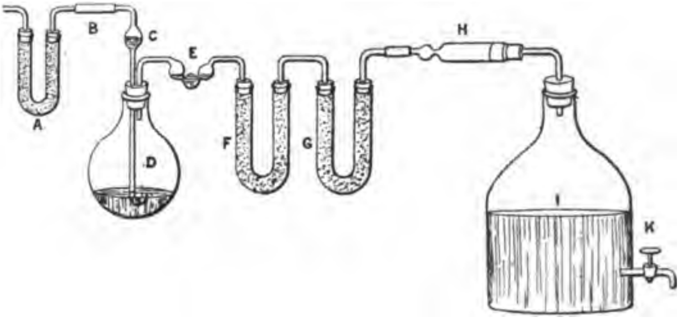

FIG. 33.

fixed, and the tube, G, accurately weighed, open the clip and allow the acid to run on to the ore in D; after which open the tap, K, and allow a gentle stream of water to flow out; a current of air thus passes through the apparatus and sweeps the CO_2 out of D, which is absorbed in the U-tube, G. Continue the aspiration in the cold until no more effervescence is observed, and then apply a gentle heat to the flask, D; continue heating to incipient ebullition until it is assured that all CO_2 is eliminated, when the tube, G, is detached and weighed. The increase in weight, $\times \frac{100}{3}$ is the percentage of combined CO_2 present.

Determination of Alumina, Manganese Oxide, Lime, Magnesia, Potash, and Soda.—Weigh out 2 grms. of sample and heat with a mixture of 20 cc. of 10 E HCl and 6 cc. of 16 E HNO_3, until all action has ceased, which may take about half an hour. Evaporate the solution to dryness and heat on sand-bath to about 130° C., for about half an hour. Cool, add 10 cc. of 10 E HCl, and heat until all that is soluble is dissolved. Dilute solution to 50 cc., filter, and wash with hot $\frac{1}{10}$ E HCl until free from Fe_2Cl_6, &c. The residue (1) is reserved. The solution is diluted to 200 cc. and 5 E Am_2CO_3 gradually added, stirring, until a slight permanent precipitate is produced. 2 cc. of 10 E HCl are now added together with 25 cc. of 5 E ammonic acetate solution, the solution diluted to 800 cc., boiled for about ten minutes, filtered, and washed with hot water. Reserve solution, dry the precipitate in a water oven, scrape into a weighed platinum crucible, ignite, cool, and weigh; the residue may contain $Fe_2O_3 + Al_2O_3 + P_2O_5$ (TiO_2). Deduct from this weight the amount of Fe_2O_3 and P_2O_5 present in 2 grms. of sample, as determined by the methods herein described, and the remainder $\times 50 =$ percentage of alumina in soluble portion. There may be some TiO_2 present in the residue; this should be tested for, and determined by, the method described on page 53, if any be present it is deducted, with the addition of the $Fe_2O_3 + P_2O_5$ already found, from the amount of residue, the remainder being the alumina. The filtrate is now poured in a 1½ litre flask, diluted to 1 litre, 3 cc. of bromine added, stirred until all has gone into solution, 20 cc. of 20 E AmHO added, the solution heated to the boil, the hydrate of manganese filtered off, washed, and estimated as directed on page 40. Evaporate the solution to about 100 cc., add 5 cc. of 5 E AmCl, 5 cc. of 20 E AmHO, and 10 cc. of E ammonic oxalate solution, stir well, and allow to stand for about ten hours. Filter off the oxalate of lime, wash with water till free from ammoniacal salts, dry, and ignite gradually in a weighed crucible, to the highest heat of the blow-lamp. Cool and weigh the residue, which is now lime. $(CaO) \times 50 =$ percentage of lime. The solution containing the magnesia and alkalies is evaporated to dryness in a platinum dish, and the residue carefully ignited until no more fumes of ammoniacal salts are given off. Cool, add 5 cc. of water, brush in about 1 grm. of oxalic acid, evaporate to dryness as before, and ignite. The alkalies are now left as carbonates, and the magnesic chloride is converted into magnesia (MgO). Treat the residue with about 20 cc. of hot water, filter off magnesia, wash with water, dry, ignite, and weigh; this weight $\times 50 =$ percentage of magnesia (MgO).

The solution is transferred to a weighed platinum dish, and the solution made acid with 10 E HCl, evaporated to dryness without loss, and the residue carefully heated to a dull red heat for ten minutes. Transfer to desiccator to cool, and weigh the $NaCl + KCl$. Add 5 cc. of water to the residue, and then sufficient E $PtCl_4$ to convert the sodic and potassic chlorides into double chlorides of the alkalies and platinum. Assuming that the whole of the residue is NaCl, a calculation of the quantity required can be made. Thus every 117 parts of NaCl require 336·38 parts of $PtCl_4$, forming $PtCl_4, 2NaCl$, 1 cc. of E $PtCl_4$ solution contains ·0841 grm. of $PtCl_4$. The mixture is evaporated (at a temperature of about 90° C.) *nearly* to dryness over a water bath; after which about 15 cc. of alcohol, sp. gr. ·86, are added, the dish covered with a glass plate, and allowed to stand for three hours, with occasional rotatory stirring of the contents; allow to settle before filtering, decant the clear liquid through a weighed filter, and finally filter off the double chloride of potassium and platinum, with the aid of the filtrate and a feather. Wash with alcohol until free from the $PtCl_4$ 2 NaCl; dry at 130° C., cool with the usual precautions, and weigh.

Calculation of the proportion of Na_2O and K_2O present:—$PtCl_4,2KCl \times \cdot 3070 = KCl$, and $KCl \times \cdot 6317 \times 50 =$ percentage of K_2O. Subtract the weight of KCl thus calculated from the weight of the $NaCl + KCl$ previously found. The remainder $\times \cdot 5302 \times 50 =$ the percentage of Na_2O present.

The reserved residue (1) is dried, brushed into a platinum crucible, 8 cc. of 10 E HCl and 4 cc. of HF added, the solution evaporated to dryness, the same quantities again added, and evaporated as before. Take up the residue in about 8 cc. of 10 E HCl, allowing to gently boil until all that is soluble is dissolved. Dilute to 30 cc., and filter if necessary. The residue, if any, must be examined for titanic acid, sulphate of barium, &c. The solution is then tested for Al_2O_3, Fe_2O_3 (TiO_2), CaO, MnO, &c., which are estimated, if present in sufficient quantities, by the foregoing methods, taking care to keep the volume of the solutions in proportion to amounts thought to be present; and the result added to those already found in the soluble portion.

Estimation of Titanic Acid (TiO_2) in Titaniferous Iron Ores.—The following process is recommended by W. Bettel.* Weigh out 0·5 grm. of the powdered sample into a platinum crucible, mix with 6 grms. of powdered and recently-fused bisulphate of potassium, and keep in a steady fusion for about twenty minutes. Allow to cool, digest for a few hours in about 180 cc. of distilled water, and filter off any silica. Dilute the solution to about 1,200 cc., and add 4 E sulphurous acid (H_2SO_3), until all the iron is reduced, and boil for about six hours, replacing the water as it evaporates. The precipitated titanic acid is now allowed to settle, the supernatant liquid decanted through a filter, and the residue washed by decantation and filtration with $\frac{E}{20}$ H_2SO_4; after which it is dried, ignited, allowed to cool, moistened with 5 E Am_2CO_3, re-ignited, cooled, and weighed. When only very small quantities of titanic acid are present, as in the case of some iron ores, a process similar to that described for the determination of titanium in iron must be adopted.

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* Crookes' "Select Methods," p. 194.

Estimation of Phosphoric Acid (P_2O_5) and Sulphuric Acid (SO_3).—Weigh out 10 grms. of the ore into a 250 cc. porcelain dish, pour on a mixture of 45 cc. of 10 E HCl and 25 cc. of 16 E HNO_3, boil until it is assured that all action has ceased, evaporate to dryness, heat on sand bath, cool, re-dissolve in 50 cc. of 10 E HCl, dilute to 100 cc., filter to about 200° C. off, wash the insoluble residue with $\frac{1}{10}$ E HCl until free from Fe_2Cl_6, pour the filtrate into a 250 cc. graduated test mixer, and dilute to 200 cc. Divide the solution in half, pouring each 100 cc. into beakers of about 150 cc. capacity. To the one add 20 cc. of 16 E HNO_3, evaporate to the consistency of syrup on water bath, dissolve in 14 cc. of 10 E HNO_3, dilute to 35 cc., add 40 cc. of ammonic molybdate reagent, stir well, allow to stand at 40° C. for three hours, filter, and determine as directed on page 39. The other 100 cc. are nearly neutralized with 5 E AmHO, heated to boiling, 10 cc. of E $BaCl_2$ added, allowed to stand for twelve hours, and the sulphur determined as directed on page 38.

The above methods are applicable only to those iron ores which are mostly smelted in this country. For the analysis of iron ores containing arsenic, vanadium, copper, pyrites, &c., special treatises must be consulted.

The following is a table of analyses of various iron ores, used for the extraction of the metal :—*

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* Thorpe, "Chemical Analysis," p. 216.

	Magnetic Ore. Dannemora.	Red Hematite. Ulverstone.	Brown Hematite. Dean Forest.	Spathic Ore. Westphalia.	Blackband Ore. Scotland.	Clay Iron Stone. Dudley.
Ferric Oxide .	70·23	94·23	90·05	2·75	2·72	0.40
Ferrous Oxide .	29·65	—	—	48·12	40·77	45·86
Manganous Oxide	—	0·23	0·88	0·83	—	0·96
Alumina . .	—	0·63	0·14	1·63	—	5·86
Lime . . .	—	0·05	0·06	1·75	0·90	1·37
Magnesia . .	—	trace	0·20	2·29	0·72	1·85
Silica . . .	—	4·90	0·92	1·62	10·10	10·88
Carbonic Acid .	—	—	—	39·92	26·41	31·02
Phosphoric Acid .	—	trace	0·09	0·54	—	0·21
Sulphuric Acid .	—	0·09	traces	—	—	trace
Iron Pyrites .	—	0·03		0·22	—	0·10
Water . . .	—	0·56	9·22	0·45	1·00	1·08
Organic Matter .	—	—	—	0·39	17·38	0·90
	99·88	100·72	100·76	100·51	100·00	100·29

MANGANIFEROUS IRON ORES.

Estimation of Manganese.—The value of these ores, largely used in the manufacture of spiegeleisen, depends on the proportion of manganese present. There are more difficulties in determining the manganese, than in ordinary iron ores, owing to the presence of baryta, zinc oxide, &c. The method adopted by Riley is as follows:—1 grm. of the dried ore is heated with 20 cc. of 10 E HCl until all that is soluble is dissolved. Dilute to 50 cc., filter off the insoluble residue, wash with $\frac{E}{10}$ HCl till free from soluble matter, dilute the filtrate to 300 cc., heat to boiling, add 5 cc. of 5 E H_2SO_4, stir well, allow to stand for four hours, and filter off any $BaSO_4$. Dilute to 1,000 cc., add 5 E Am_2CO_3 gradually, until a slight but permanent precipitate is produced; dissolve this in 3 cc. of 10 E HCl, add 20 cc. of 5 E ammonic acetate, boil for ten minutes, filter, dissolve the unwashed precipitate.

which may contain some manganese, in 10 cc. of 10 E HCl, re-precipitate the iron as basic acetate as before, with Am_2CO_3 and $AmC_2H_3O_2$ and filter. Mix the filtrate, evaporate down to 1,200 cc., add 3 cc. of bromine, precipitate the manganese as $Mn(HO)_2$ with 20 E AmHO, and estimate as directed on page 40. The ignited Mn_3O_4 should always be examined for impurities, such as baryta, zinc oxide, and lime.

Analysis of Limestones, Boiler Incrustations, and Siliceous Substances.—Weigh out 1 grm. of sample, and dry in a platinum crucible of 30 cc. capacity at 100° C. until no furthur loss occurs. Loss = free moisture. Add 4 grms. of white flux ($K_2CO_3 + Na_2CO_3$), fuse over blow-lamp for ten minutes, allow to cool, put the crucible and its contents into a 200 cc. beaker, pour on about 80 cc. of hot distilled water, and stir with a glass rod until the fused mass is detached from the crucible; lift the crucible out of the liquid with a pair of bone forceps, and wash it with hot water. The solution is now boiled, the precipitate allowed to settle, the clear liquid decanted through a filter, 50 cc. of hot water poured on the residue, boiled, and filtered as before; this operation is continued until the washings scarcely turn red litmus paper blue. The precipitate then is filtered off and washed completely with boiling water on the filter. The solution is acidulated with 10 E HCl, evaporated to dryness on a sand bath, cooled, 50 cc. of E HCl poured on to the residue and heated until all that is soluble is dissolved. It is then filtered, the insoluble residue washed till free from chlorides, the solution diluted to 100 cc., heated to boiling, E $BaCl_2$ added until no further precipitation takes place, allowed to stand twelve hours, and the $BaSO_4$ filtered off and estimated. The silica is dried, ignited in a weighed platinum crucible, cooled and weighed. It should be tested for impurities, such as TiO_2, Al_2O_3, &c., by evaporation with H_2SO_4 and HF, and if any residue is left it is to be examined and taken into account. The bases left on the filter, which may contain CaO, MgO, Fe_2O_3, Al_2O_3, are dissolved off with about

10 cc. of 10 E HCl, with addition of a few crystals of $KClO_3$, the filter is washed with water, 5 cc. of 5 E $AmCl$ added and the solution diluted to 100 cc. 20 E $AmHO$ is now added until the solution is just alkaline, boiled, and the Fe_2O_3 and Al_2O_3 filtered off and determined by the method given on page 89. E ammonic oxalate is now added to the filtrate until no further precipitation takes place, stirred, the calcic oxalate allowed to subside, after which it is filtered off, and the CaO estimated as directed on page 112. E sodic phosphate is added to the filtrate until no further precipitation takes place (if there be no immediate precipitate about 10 cc. will be found sufficient), allowed to stand at least twelve hours, the ammonio-magnesic phosphate filtered off, the MgO determined as described on page 204, the potash and soda may be estimated as described on page 112, and the CO_2 estimated as described on page 110.

The composition of a boiler incrustation will depend upon the heat that it has been subjected to. That part which is nearest the iron and the heat will contain less carbonic acid than that on the surface, owing to its being driven off by the heat from the carbonate of lime and magnesia present, with the formation of caustic lime and magnesia, which eventually become converted into hydrates as the boiler cools down. In order to determine the combined water, about 1 grm. is ignited over a blow-lamp until it ceases to lose weight. By this means the combined water and carbonic acid and free moisture are driven off, and by subtracting the amount of carbonic acid and free moisture from the loss, as found in another portion, the proportion of combined water is obtained.

The following is an analysis of a boiler scale by the above process by Stillman :—

	Per cent.
Silica and Clay	11·70
Ferric Oxide and Alumina	2·81
Sulphate of Lime	1·69
Carbonate of Lime	5·45
Carried over	21·65

	Per cent.
Brought forward	21.65
Carbonate of Magnesia	7·36
Calcium Hydrate (CaO, H_2O)	13·70
Magnesium Hydrate ($MgOH_2O$)	56·37
Moisture driven off at 100° C.	0·69
Undetermined	0·20
	99·97

By a slight modification many other substances can be analysed by the above process, such as clays, fire-bricks, furnace slags, &c.

ANALYSES OF SILICEOUS MATERIALS.

	Glenboig Firebrick.	Newcastle Firebrick.	Dowlais Firebrick.	Sheffield Ganister.
Silica	62·10	58·00	97·5	89·04
Alumina	33·10	36·50	1·4	5·44
Ferric Oxide	3·00	1·67	0·55	2·65
Lime	0·90	0·50	0·15	0·31
Magnesia	trace	0·90	0·10	0·17
Potash	0·90	2·12	—	—
Soda	—	·30	—	—
Loss on Ignition	—	—	—	2·30
	100·00	99·99	99·70	99·91

ANALYSES OF VARIOUS CLAYS (DR. MILLER).

	Washed Kaolin.			Stourbridge Fire Clay.	Pipe Clay.	Sandy Clay.	Blue Clay.	Brick Clay.
	Chinese	St. Yrieix.	Cornish.					
Silica	50·5	48·37	46·32	64·10	53·66	66·68	46·38	49·44
Alumina	33·7	34·95	39·74	23·15	32·00	26·08	38·04	34·26
Ferric Oxide	1·8	1·26	0·27	1·85	1·35	1·26	1·04	7·74
Lime	—	—	0·36	—	0·40	0·84	1·20	1·48
Magnesia	0·8	trace	0·44	0·95	trace	trace	trace	5·14
Potash and Soda	1·9	2·40	12·67	—	—	—	—	—
Water	11·2	12·62		10·00	12·08	5·14	13·57	1·94
	99·9	99·60	99·80	100·05	99·49	100·00	100·23	100·00

AVERAGE ANALYSIS OF BLAST FURNACE SLAG. (THORPE)

Silica	41·85
Alumina	14·73
Ferrous Oxide	2·63
Manganous Oxide	1·24
Lime	30.99
Magnesia	4·76
Potash	1·90
Calcium	1·15
Sulphur	0·92
Phosphoric Acid	0·15
	100·32

A GOOD MIXTURE FOR LINING A "BASIC" BESSEMER CONVERTER.

Magnesia	37
Lime	51
Silica	8
Alumina and Oxide of Iron	4
	100

ANALYSES OF MILL-FURNACE SLAGS.

	Dowlais.	Wasseral-fingen.	Sweden.	—
	Riley.	Rammels-berg.	Dugent.	Noad.
Ferric oxide	—	8·49	—	5·00
Ferrous oxide	66·01	55·36	65·83	52·50
Manganous oxide	0·19	—	0·74	—
Alumina	2·47	—	—	9·60
Lime	0·81	0·36	—	—
Magnesia	0·27	trace	trace	—
Silica	28·71	34·00	33·47	—
Sulphur	0·11	—	—	32·00
Ferrous sulphide	—	—	—	1·95
Phosphoric acid	1·22	—	—	0·25
Copper	trace	—	—	—
	99·79	98·21	100·04	101·30
Iron, per cent.	51·34	49·0	51·20	45·34

PART III.

FUELS—SOLID, LIQUID, AND GASEOUS.

AT a time when fuel has become such an expensive commodity, the consideration of its quality, heating power, and economic application, becomes a matter of great importance to engineers and steam users in general.

Fuel in the unrestricted sense embraces all substances capable of burning in the air, with the production of heat. The amount of heat given out, however, varies enormously with the kind and chemical composition of the substance used. The substances usually regarded as *fuel* have generally a pure organic or vegetable origin, and include all kinds of coal, peat, turf, wood, coke, charcoal, patent fuels, &c.; and also combustible gases, liquid fuel (hydrocarbons, &c.). Now all these fuels mainly consist of more or less carbon and hydrogen, which are the essential heat-producing elements, together with oxygen, nitrogen, sulphur, phosphorus, and ash. The more organic oxygen that is present in a fuel the less heat will be given out, owing to its already being in chemical combination with carbon or hydrogen. Sulphur and phosphorus are very undesirable elements in fuel, especially when the fuel is used for such metallurgical operations as iron smelting. Generally speaking the more ash a fuel contains the less will be its calorific value, and more inconvenience and loss of heat will be brought about by the formation of "clinkers," &c. Where enormous quantities of fuel are used, such as on railways, steamboats, ironworks, &c., the quality, it will be seen at once, becomes an important question, especially now since such a high price has to be paid for it.

In determining the value of a solid fuel such as coal, coke, and patent fuel, it is necessary to estimate the amount of moisture, volatile matter, coke and its quality, sulphur, the amount of ash left on the incineration of sample, together with the calorific and evaporating power ascertained—(1) Theoretically, from a knowledge of the percentage of hydrogen, carbon, and oxygen found to be present, by an elementary organic analysis of the substance; or (2) Practically, by igniting a known weight in a calorimeter in oxygen, and ascertaining the amount of ice that has been melted, or observing the increase in temperature of a known weight of water; or (3) By ascertaining the amount of lead reduced from its oxide, by a known weight of the sample. This last method, however, is not often adopted for calorific power, the amount of lead reduced being a measure of the reducing power of a sample, rather than the amount of heat that it is capable of evolving.

The calorific and evaporating power of fuels, estimated by the foregoing methods, must only be taken relatively. The actual highest practical value of any fuel would depend very materially upon the kind of furnace used, and the regulation of an appropriate influx of air, so as to insure complete combustion. The heat carried up the flues by the products of combustion should, if possible, be reduced to a minimum, so as to obtain the most economic results.

Liquid fuels are now coming to the front, and, judging from recent experiments made on locomotives in South Russia, where petroleum is plentiful and cheap, and coal comparatively dear, they seem to have the advantage over coal. The experiments made in this country, notably on the Great Eastern Railway, with liquid fuel on locomotives, and also on steamboats, have given very encouraging results, the great difficulty, however, to contend with, being a scarcity of oils at a price that would favourably compete with coal; and, moreover, even if a good supply of liquid fuel were available, the chances are that its market value would soon be risen considerably higher than the price that could be afforded to be given for it. The specific gravity, flashing point, and the calorific power obtained practically by slightly modifying the process as used in the case of coals, or by calculating it theoretically from the percentage of carbon and hydrogen found to be present, would be valuable data for guidance as to their selection. Sulphur, generally speaking, is present in so small a quantity as to be not worth consideration.

With regard to gaseous fuel, the principal heat-giving constituents usually present are hydrogen, gaseous hydrocarbons (marsh gas, olefiant gas, &c.), and carbonic oxide. The determination of these, and the calculation of their respective calorific values on combustion, would convey an estimate of their efficiency for heating purposes.

Estimation of Moisture and Ash (Solid Fuels).—Weigh out 3 grms. of the fairly averaged powdered sample in a shallow platinum dish, dry in an air bath regulated to 105° C. for one hour, allow to cool in a good desiccator, and weigh; the loss is taken as moisture.

Coal and cokes should not be allowed to dry for more than one hour, otherwise the result obtained for moisture will be too low, owing to the oxidation by the air of the pyrites present.

The dish with the dried sample, is now cautiously heated to redness in a muffle, until it is seen that all carbonaceous matter has been burnt off, and then allowed to cool in the desiccator and weighed. It is once more heated in a muffle for about five minutes, and again cooled and weighed. If the weight is unaltered from that of the last weighing, the increase in weight over the dish, is the amount of ash left by 3 grms. of sample, which can easily be expressed centesimally. The amount of ash left by different samples is very variable, some coals showing only 2 per cent., while others as much as 68 per cent.

The following shows the variation in the amounts of ash that may be present in different coals:—

Bituminous Coals	1·7 to 33·9 per cent.
Semi-bituminous Coals . .	2·8 to 23·7 ,,
British Coals	1·2 to 26·5 ,,
Anthracites, American . .	1·3 to 68·0 ,,

For proportion of ash found in various kinds of coals see table on page 180.

It is needless to say that the greater the amount of ash left by a coal, the lower will be its calorific value and the more inconvenience will be caused, resulting from formation of clinkers, &c. The composition of the ashes of coals varies considerably with the nature of the coal and the locality in which it is found, the coal generally containing the same substances as the surrounding geological strata. The ash of bituminous coal consists, to a great extent, of silicate and sulphate of lime, together with oxide of iron and argillaceous matter; while the ash of the cannel class of coals consists largely of silicate of alumina. For the composition of ashes left by various coals see page 179, and for methods of analysing ash, page 166.

Chemical Classification of Coal.—At a meeting of the South-Wales Institute of Engineers held at Cardiff on April 30th, 1900, Mr. Clarence A. Sayler, B.Sc., F.I.C., gave a valuable paper on the "Chemical Classification of Coal," and after reviewing the various classifications in vogue, and commenting upon ambiguous local terms such as "dry," "hard," "strong," "fat," "half-fat," and "high-fat," bituminous and semi-bituminous, he proposed to adopt a classification on scientific principles as follows:—

I. *Anthracitic carbon plane.* Carbon over 93·3 per cent.
  (*a*) Ortho hydrous.—Hydrogen under 4 per cent., volatile matter under 9 per cent. (5 to 9 per cent.). Ortho-anthracite.
  (*b*) Per-hydrous.—H. over 4, V. M. over 9 (9-15). Semi-anthracite.

II. *Carbonaceous plane.* C. 93·3-91·2.
  (*a*) Sub-hydrous.—H. under 4·2; (1) V. M. 7·7-12. Sub-carbonaceous. (2) V. M. under 7·7-12. Sub-pseudo-anthracite.
  (*b*) Ortho-hydrous.—H. 4·2-4·45, V. M. 10-14. Carbonaceous.
  (*c*) Per-hydrous.—H. over 4·45, V. 14-24. Semi-bituminous.

III. *Hydro-carbonaceous or bituminous plane.* C. 91·2-84.
  1. Meta-bituminous. C. 91·2-89.

(*a*) **Sub-hydrous.**—H. 4·5-4·9, V. M. 16-23. Sub-meta-bituminous. H. 3·7-4·5, V. M. 10-16. Sub-pseudo-carbonaceous. H. under 3·7, V. M. under 10. Sub-pseudo-anthracite.
(*b*) **Ortho-hydrous.**—H. 4·9-5·7, V. M. 23-30.
(*c*) **Per-hydrous.**—H. over 5·7, V. M. 30-44.

2. **Ortho-bituminous.** C. 89-87.

(*a*) **Sub-hydrous.**—H. 4·5-5, V. M. 16-23. Sub-ortho-bituminous. H. under 4·5, V. M. under 16. Sub-pseudo-carbonaceous.
(*b*) **Ortho-hydrous.**—H. 5·0-5·7, V. M. 23-30.
(*c*) **Per-hydrous.**—H. 5·7 and over, V. M. over 36 (seldom over).

3. **Para-bituminous.** C. 87-84.

(*a*) **Sub-hydrous.**—H. 5·0 and under, V. M. Sub-para-bituminous.
(*b*) Ortho-hydrous.—H. 5·0-5·8, V. M. 30-40 (usually).
(*c*) Per-hydrous.—H. over 5·8, V. M. over 40 (seldom under).

IV. *Carbhydratous or lignitious plane.* C. 84-75.
Meta-lignitious, C. 84-80; ortho-lignitious, C. 80-75.
(*a*) Ortho-hydrous.—H. 4·7-5·8 }
(*b*) Per-hydrous.—H. over 5·8 } V. M. 31·57.

Classification of Carbonaceous Coal according to Volatile Matter.—Coals of any carbon class may be classified according to volatile matter as follows:—

Volatile matter	up to	8	per cent.	*anthracite* and *pseudo-anthracite.*
,,	,,	8 to 16	,,	*carbonaceous* and *pseudo*-carbonaceous.
,,	,,	16 to 26	,,	short-flame coals (mostly semi-bituminous or sub-bituminous).
,,	,,	26 to 32	,,	normal bituminous.
,,	,,	over 32	,,	large-flame coals.

Brown Coal from Gippsland.—Although somewhat out of place here (see *post* pp. 175—183), the following particulars of an analysis (by the Author) of Brown Coal from Gippsland, Australia, may be mentioned:—

SAMPLE DRIED AT 230° F.

	Per cent.
Carbon	67·59
Hydrogen	5·27
Oxygen	23·05
Nitrogen	1·28
Sulphur	0·25
Ash	2·56
	100·00
Moisture	20·55
Tar and gas	48·54
Coke	51·47
Fixed carbon	48·92
Specific gravity	1·25

Estimation of Coke and Volatile Matter.—1 grm. of the sample is carefully weighed into a small porcelain crucible provided with a cover, cautiously heated to redness by a large Bunsen burner for two minutes, and then heated for an additional two minutes at a higher temperature over a gas blowpipe; it is then allowed to cool in a desiccator and weighed. The loss = moisture + volatile matter ; and the residue = coke (fixed carbon + ash).

The coke is tested by means of a penknife, to ascertain if it is friable or compact. A little of it is placed on platinum foil and ignited, and noted as to whether it burns freely or not.

Mr. G. E. Davis makes an interesting classification of coals, according to the amount of coke they are capable of producing, as follows:—

(1) Splint coal, burning with a long flame, and yielding from 50 to 60 per cent. of powdery or slightly caked coke.

(2) Gas coal, or coal of a bituminous nature, burning with a long flame, and yielding from 60 to 70 per cent. of fused but deeply seamed coke.

(3) Smithy coal, or true bituminous coal, burning with a long flame, and yielding 68 to 75 per cent. of fused compact coke.

(4) Caking coal, burning with a short flame, leaving from 75 to 82 per cent. of fused compact coke.

(5) Anthracite or smokeless steam coal.

Estimation of Sulphur.—Sulphur is a very deleterious constituent of coal and coke, both for boilers and metallurgical purposes. A very small quantity finding its way into "pig" iron, will render the latter unfit for steel-making, owing to its producing "red-shortness," while the sulphurous vapour formed by its combustion in boilers, corrodes fire-boxes, boiler-tubes, &c.

The disagreeable odour in our underground railway tunnels, is largely attributable to compounds of sulphur (sulphuretted hydrogen, bisulphide of carbon, &c.), mainly brought about by the distillation of fresh portions of coals, thrown on to the red-hot coke in the fire-boxes of the locomotives.

Sulphur exists in three forms in coals and cokes, being present as iron pyrites (FeS_2), technically termed "brasses," and sulphate of lime ($CaSO_4$), and combined organically with carbon and hydrogen.

The sulphur, present as iron pyrites, and that organically combined, alone appear to affect the economic application of the fuel.

To determine the total sulphur present, weigh out 2 grms. of the sample and mix thoroughly with 5 grms. of pure powdered nitrate of potash, and add this mixture in small portions at a time, to 8 grms. of pure anhydrous sodic carbonate kept in a steady state of fusion in a capacious silver crucible by means of the oxidizing flame of a Bunsen burner. The crucible should be tilted, and the flame kept as far from its mouth as possible, to prevent access of sulphur compounds, from the gas. When the mixture has been fusing for about ten minutes, after the addition of the last portion of the sample, it is acidulated with dilute hydrochloric acid, evaporated to dryness, heated on sand bath to about 130° C. to render silica insoluble, moistened with 10 cc. of 10E HCL, diluted with distilled water to 100 cc., filtered and washed, and the filtrate diluted to about 400 cc., and then heated nearly to boiling. A few cc. of a saturated solution of baric chloride are now added, the solution well stirred, and then allowed to stand in a warm place, if possible, for about twelve hours. The supernatant liquid is now syphoned off, and the precipitated baric sulphate ($BaSO_4$) carefully filtered through a No. 2 Swedish filter paper, and washed till free from soluble matter with hot water. The filter paper is then spread out on a watch glass, and put to dry in a water oven: in the meantime a small porcelain crucible is carefully weighed. When the filter is dry, the $BaSO_4$ is brushed into it, the filter burnt separately, and its ash added to the main portion. The crucible and its contents are then ignited at a dull red heat in the oxidizing flame, allowed to cool, and weighed. Increase in weight = $BaSO_4$ + ash. Subtract ash, then $\frac{BaSO_4 \times \cdot 1373 \times 100}{2} =$ percentage of total sulphur.

The total sulphur can also be conveniently estimated from the solution left on the determination of the heating power by the calorimeter described on page 135. All that has to be done is to acidulate the solution with 10E HCL, filter off matter that has escaped combustion, evaporate, filtrate to about 400 cc. in a porcelain basin, and precipitate sulphur with $BaCl_2$, and determine as above described.

The sulphur present as sulphate of lime is determined by boiling 5 grms. of sample with a strong solution of sodic carbonate; the sulphur is by this means converted into soluble sulphate of soda; acidulate with HCL, dilute and filter, and estimate sulphur as above. On subtracting the sulphur thus found from the total, the amount of sulphur present as pyrites is obtained.

A somewhat new method for the determination of the total sulphur in cake is described by Neilson in the *Chemical News*, April 24th, 1891. The details of the process are as follows:—1 gram of the coke is intimately mixed with 2 grams of anhydrous carbonate of soda ($Na_2 CO_3$) and 0·5 gram of carbonate of manganese ($Mn CO_3$), and the whole brushed into a flat-bottomed platinum capsule and kept at a dull red heat for one hour. After this time the whole of the sulphur and carbon will have been oxidized. The mixture is then carefully fused for about 3 minutes, cooled, and the whole placed into a 100 cc.

beaker, and 40 cc. of dilute HCL (20 cc. 10E HCL and 20 cc. water) poured on it. The mixture is now heated until all that is soluble is dissolved, the capsule taken out with a pair of platinum or bone forceps, and washed down with water from a wash bottle, into the beaker. The solution is now evaporated to dryness, and the residue heated for a short time at about 130° C. on a sand bath to render silica insoluble, cooled, warmed with about 5 cc. of strong hydrochloric acid, diluted to 200 cc. with distilled water, the insoluble residue filtered off, and the sulphur estimated in the filtrate, by precipitation as $BaSO_4$ with $BaCl_2$ in the usual manner.

Hundeshagen method for the determination of Sulphur in Fuels.—The writer has had considerable experience with this process, and is quite satisfied with the accuracy of the results. The mixture used in this method is one part of pure and dry potassium carbonate to 2 parts of magnesium oxide. To 1 part of coal at least 2 parts of the mixture is used, three-fourths of the reagent is intimately mixed with the finely powdered coal and the remaining one-fourth is spread over the top; the combustion is complete if the mixture is kept at a red heat for half an hour. The rest of the process is conducted as described on page 124.

The amount of sulphur in coals varies from about 0·2 per cent. to as high in some cases as 10 per cent., a fair average in British coals being about 1·5 per cent. In the coking of coals something like 50 per cent. of the sulphur combines with hydrogen and carbon in the coals and escapes as gas in the form of bisulphide of carbon, sulphuretted hydrogen, &c. Coals or cokes containing iron pyrites, if heated to a high temperature in presence of water, give off considerable quantities of sulphuretted hydrogen, due to the decomposition of the pyrites with the formation of oxide of iron. Apart from the detrimental influence of iron pyrites in coal and coke on iron smelting, &c., it causes much inconvenience and loss of fuel on locomotives, &c., owing to the combination of the resulting oxide of iron with the silica of the ash, forming a fusible slag or clinker which covers the bars in the firebox, thus preventing free access of air and causing the formation of carbonic oxide instead of carbonic acid, which consequently results in a loss of much heat.

There have been numerous experiments tried with a view of desulphurizing coal and cokes, such as mixing common salt with coal previous to coking, heating coke to redness in a current of steam, coking with salt and washing, heating the coke below a red heat in a current of air under pressure, mixing the coal with such substances as lime, chalk, soda, oxide of manganese, &c., previous to coking, &c., but, unfortunately, none of these processes have resulted in eliminating sufficient sulphur to warrant their adoption on a large scale.

Estimation of Nitrogen.—Owing to the small quantity of nitrogen usually present in fuels, it is best determined volumetrically. The following is the process devised by Dumas. A combustion tube is selected about 120 cm. long, sealed at one end like a test tube, cleaned, and dried. A layer of pure bicarbonate of soda 15 cm. long is first introduced, then a layer of copper oxide 20 cm. long, after this an intimate mixture of 1·5 grm. of the sample with oxide occupying 30 cm., then 30 cm. of coarse copper oxide, and 20 cm. of small copper turnings; the tube is now connected by means of a good fitting cork with the bent delivery tube, B, Fig. 34, and placed in a combustion furnace. The further end of the tube containing the bicarbonate is then gradually heated to the extent of 6 cm. Carbonic acid gas is evolved and sweeps the air out of the tube; when the gas has been coming off for a few minutes, the end of the delivery tube is dipped under mercury contained in the trough, M, and the issuing gas tested for air, by inverting a test tube filled with a 10 E solution of potassic hydrate over it. If the gas as it comes in is completely absorbed, all air has been eliminated; if not, continue the heating until the desired point is attained. The graduated tube, A, which is filled ⅔ with mercury and ⅓ with a 10 E solution of potassic hydrate, is then inverted over the end of the delivery tube and held in position by the clamp, K. The combustion is now proceeded with. The fore part of the tube containing the copper is first cautiously heated to redness, and the heat gradually extended to the further end until the point where the sample ends. When no more gas comes off from the sample, the other half of the bicarbonate is heated; the second crop of CO_2 thus produced drives any nitrogen still left in the tube into A. When the volume of gas in the tube, A, is no longer diminished by the absorption of any CO_2 that may still be present, even on shaking, the tube is transferred, by means of a small dish filled with mercury, to a deep vessel containing water. The mercury will then be displaced by water; the tube is pushed into the water until the levels of the liquids are coincident, when the volume of nitrogen is noted, together with the temperature and barometric pressure. The volume observed is reduced to 0° C. and 760 mm. pressure; and inasmuch as the gas is measured over water, allowance must be made for the pressure reduced by the tension of aqueous vapour at the temperature observed.

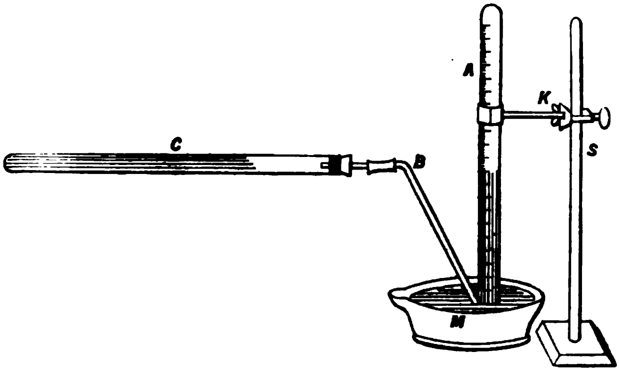


FIG. 31.

The following is an example of an actual determination of nitrogen in a sample of South Stafford coal:—

Volume of nitrogen observed	25 cc.
Temperature	15° C.
Barometric pressure	758 mm.

The tension of aqueous vapour at 15° C. is equivalent to 12·677 mm. of mercury.

Taking 1 cc. of nitrogen at 0° C. and 760 mm. pressure as weighing ·0012544 grm., then the percentage of nitrogen by weight in the sample is—

$$\frac{25 \times 273 \times (758 - 12{\cdot}677) \times {\cdot}0012544 \times 100}{(273 + 15) \times 760 \times 1{\cdot}5} =$$

1·943 per cent. nitrogen.

The amount of nitrogen normally present in coals varies from about 0·5 to 2·5 per cent., and under proper conditions of destructive distillation, practically the whole of it combines with hydrogen with the formation of ammonia. In the manufacture of coal gas the ammonia finds its way into the aqueous liquor, where it exists partly "free" and partly "combined," from which it is obtained by distilling with lime; and by passing the gas through sulphuric acid, sulphate of ammonia is obtained, which is now much used as a manure, &c.

Estimation of Carbon and Hydrogen.—The principle of the method adopted for the determination of carbon and hydrogen depends upon the fact that when a fuel is burnt in excess of air or oxygen, or any oxidizing substance, the carbon is oxidized into carbonic acid gas (CO_2), while the hydrogen is converted into water (OH_2), evolved as steam. A known weight of the sample being taken, it is ignited with chromate of lead, or with copper oxide and oxygen or air; the products of combustion, CO_2 and OH_2, are absorbed by appropriate reagents, and weighed separately, from which the amounts of carbon and hydrogen present are deduced by a simple calculation.

Select a wrought-iron tube 20—22 mm. in diameter and 115 cm. long; oxidize inner surface of tube by heating it to redness in a combustion furnace, and passing a current of steam through.

A layer of recently ignited coarse oxide of copper, about 20 cm. long, is pushed into the middle of the tube and kept in position by plugs of copper gauze, placed one each side; a sheet-iron boat about 30 cm. long, is nearly filled with recently fused and powdered chromate of lead, and introduced into the tube, which is placed in a combustion furnace and heated below the fusing point of the chromate, a current of dry air being passed through, to rid the tube of any moisture. The gas is then put out and the tube plugged and cooled. The boat is then taken out, and from ·3 to ·5 grm. of the powdered sample, free from water, quickly and thoroughly mixed with the chromate, and replaced in the tube; a similar boat filled with recently reduced metallic copper is introduced at the other end of the tube, and the whole placed into the furnace and coupled up to the desiccating and absorption apparatus. Fig. 35 shows the apparatus ready for a combustion.

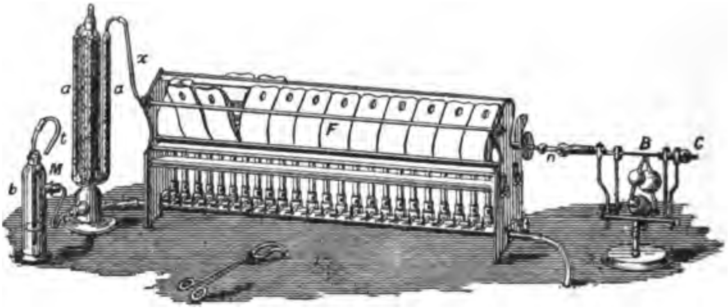


Fig. 35.

b is a cylinder containing a strong solution of potassic hydrate (10 E), which absorbs the great bulk of CO_2 present in the air used for the combustion; it is coupled up to a gas holder by means of the tube *t*, and to the cylinder, *k*, which is filled with fragments of soda lime, which completes the absorption of CO_2. The U tubes *a a*, are filled with dry granulated calcium chloride, which completely absorbs atmospheric moisture. They are connected to the combustion tube by means of the pipe, *x*; the bulb tube, *n*, contains dry calcic chloride which serves to absorb the water brought about by the combustion of the hydrogen in the sample. The bulbs, B, contain a strong solution of potassic hydrate, which absorbs the CO_2 produced by the combustion of the carbon. *c* is a small tube containing calcium chloride, which serves to retain any moisture carried mechanically from B by the issuing gas.

Before proceeding with the combustion, *n* and B are accurately weighed separately and attached to tube as in sketch; all joints having been insured air tight, the gas is turned on at the end of the tube containing the reduced copper, and a gentle current of air made to pass through the apparatus, when it is at a dull red heat. The gas is then gradually turned on until the boat containing the sample is reached. Care is here required, and the heat should be gradually raised to full redness, when the chromate will fuse, and the sample is soon completely oxidized. After it has been insured that the combustion is complete, the calcium chloride tube, *n*, and potash bulbs, B, are detached and weighed. The increase in weight of the $CaCl_2$ tube, multiplied by ·1111, gives the amount of hydrogen in quantity of sample taken, and the increase in weight of the bulbs, B, multiplied by ·27273 gives the amount of carbon present in the quantity of sample taken.

In estimating the carbon and hydrogen in non-volatile liquid fuels, the two boats are filled with copper oxide, the weighed liquid absorbed in one of the boats, and the combustion proceeded with as above; or if it be a volatile hydrocarbon it is weighed into a small tin tube, with a loose stopper, and dropped into the boat, due care being taken that the combustion is not hurried, otherwise vapour will escape unoxidized.

The following is an example of the results obtained for carbon and hydrogen in a Scotch bituminous coal, when ·5 grm. was taken :—

Weight of $CaCl_2$ tube before combustion . . .	30·4562
" " after " . . .	30·6884
Water absorbed	·2322

·2322 × ·1111 × 2 × 100 = 5·159 per cent. hydrogen.

Weight of potash bulbs before combustion . . .	52·0318
" " after " . . .	53·4296
CO_2 . .	1·3978

1·3978 × ·27273 × 2 × 100 = 76·244 per cent. carbon.

The higher the proportion of hydrogen present in fuel, the more gas in the form of combustible hydrocarbon is given off, and consequently the more flame will the fuel produce on combustion. It is on this account that wood, peat, cannel coals, &c., are so readily ignited. On the other hand, charcoal, anthracitic coal, and coke, &c., which contain such a small proportion of hydrogen, and mainly composed of carbon, ignite with much difficulty, and require more air locally to effect their complete combustion. The proportion of hydrogen varies considerably in different coals. While some anthracites contain only 1·5 per cent., Boghead cannel coal contains as much as 8·86 per cent.

The proportion of carbon varies considerably even in the same classes of coals. Some anthracites of France contain only 70·2 per cent. of carbon, while the South Wales variety contains as much as 94·2 per cent. of this element.

The proportion of carbon in cannel coal varies from 56·8 to 83·8, and in the bituminous coals from 75 to about 83 per cent.

Estimation of Oxygen.—There is no ready method for the direct determination of oxygen in complex organic compounds. It is as a rule estimated by difference. Having a knowledge of the percentage of all other ingredients present in the sample, add these up and subtract from 100, and the remainder may be taken as oxygen.

The proportion of organic oxygen present in coal varies from practically none in some kind of anthracites to as much as 15·5 per cent. in some of the Scotch coals. Generally speaking, the more organic oxygen there is in a fuel the less heat is given out, owing to its being already chemically combined with carbon or hydrogen, thus lowering the calorific value.

Specific Gravity of Coal and Coke.—It is often desirable to know the amount of space that a given weight of coal will occupy, and the determination of the specific gravity thus becomes necessary.

To obtain this a small flask provided with a thermometer stopper, and holding a definite weight of water at a known temperature—usually 60° F., which is previously accurately ascertained—is taken, and 2 to 3 grms. of the sample weighed into it; water is then added, and the coal allowed to soak in it for some time, so as to eliminate air from the pores. The bottle is then filled with water at the standard temperature and again weighed.

The specific gravity is obtained as follows:—

Let $W =$ weight of sample in air
$R =$ weight of flask $+$ water
$R_1 =$ weight of flask $+$ water $+$ sample

Then,

$$\text{Specific gravity} = \frac{W}{W + R - R_1}$$

The weight of a cubic foot of the sample in pounds is obtained by—

$$\text{log. specific gravity} + 1{\cdot}79588 = \text{log. weight of cubic foot.}$$

The number of cubic feet in a ton

$$= 1{\cdot}55437 - \text{log. specific gravity} = \text{log. cubic feet.}$$

It is very important in determining the specific gravity of coal and coke, to insure that all air has been driven out of the sample by the water before diluting and weighing. An example may be given of the error that would be involved if this precaution were not taken. Mr. Crookes, F.R.S., obtained the following: 2·76 grms. of coal gave a specific gravity of 1·309 at 64° F., immediately after filling the flask with water; after soaking twelve hours the specific gravity had increased to 1·328 for the same temperature. So that the latter determination would make a cubic foot of this coal weigh 82·76 lbs., and the former only 81·58, or 1·18 lbs. less.

The specific gravity of coals varies from 1·200 to 1·900. In many coals the specific gravity increases with the proportion of ash, although this is not a general rule.

Specific Gravity of Liquid Fuels.—The specific gravity of liquid fuels can in the majority of cases be determined at 15° C. by the hydrometer in the usual manner. It sometimes happens, however, that liquid fuels are too thick to obtain an accurate result by this means, and it has to be determined by the specific gravity bottle; or if too thick for this, by placing a drop in a cylinder of alcohol at 15° C., and adding water until it remains stationary in any part of the fluid in which it is placed, a glass rod being used for the purpose. The specific gravity of the fluid is then taken with the hydrometer, and the specific gravity of the sample is thus attained.

The Flash Point of Liquid Fuel.—The temperature at which the vapour coming off from liquid fuels ignites when mingled with air, on the application of a flame, is of some importance. The lower the temperature at which the vapour is capable of igniting, the more care, of course, will be required with its handling, storage, transport, &c.

A ready mode of determining the flash point of a sample is to pour some of the liquid into a beaker (2″ × 2″) to within about half-an-inch from the top; then cover with a disc of asbestos, through which a thermometer passes to within a quarter of an inch from the bottom of the beaker. The beaker, &c., is now put into a shallow sand bath, and surrounded with sand to the level of the liquid; a small flame is then applied under the bath, and the temperature allowed to rise about 2° per minute. After each rise of 1°, the asbestos disc is turned to one side, and a flame is quickly put into the vapour. The temperature at which it ignites is taken as the flash point.

A more definite method of testing the flash points of light oils is obtained by Sir Frederick Abel's standard flash-point apparatus described on page 262.

Determination of the Calorific Value of Solid and Liquid Fuels by Thompson's Calorimeter.—In this method, which is now most extensively used in estimating the heating power of fuels, a known weight of sample is ignited with an oxygen mixture in a copper cylinder, in a known weight of water, the temperature of which is first accurately observed. From the increase of temperature of the water, due to the combustion of sample, the comparative heating and evaporating power can be deduced.

The French unit of heat, is the amount of heat necessary to raise the temperature of 1 grm. of water through 1° C., or more correctly from 0° to 1° C. The calories as here expressed are the number of grms., lbs., or any unit weights of water raised 1° C. by the combustion of 1 grm. or 1 lb., &c., of the sample. These can be converted into British thermal units—viz., lbs. of water raised 1° F. by 1 lb. of sample—by multiplying by $\frac{9}{5}$.

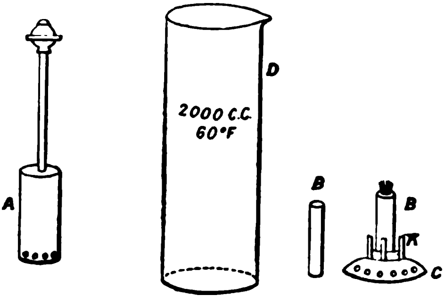
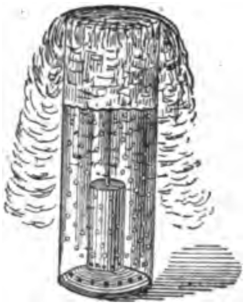


Fig. 36.



Calorimeter in Action.

For coals and cokes, 2 grms. of the finely powdered and dried sample, are thoroughly mixed with 26 grms. of a finely powdered and dry mixture, of chlorate of potash, 3 parts, and nitrate of potash, 1 part, on a sheet of glazed paper. By means of a flexible steel spatula, the mixture is introduced in small quantities at a time into the copper cylinder B, Fig. 36, each addition being pressed with about the same pressure, with the rounded end of a test-tube, so that a fairly uniform combustion can be relied upon. When all has been brushed in, a short length of fuse (prepared by soaking lamp cotton in a strong solution of nitre, and drying) is pushed into the mixture, and about half an inch allowed to protrude. It is then fixed into the brass stand C. The glass cylinder D, having been charged with 2,000 cc. of water, the condenser A is fixed over B into C, held firmly by the clutches K. The whole is then placed into D, and moved up and down in the water until the temperature of the latter is fixed. The temperature is recorded by a very delicate thermometer graduated to $\frac{1}{10}$ of a degree Centigrade.

The temperature of the room is usually higher than the water, so that a little hot water or ice (as the case may demand) must be added, until the differences are about as follow:—

Room at ° C.	27	23	20	16	13	10	6
Water should be	21	18	15	12	10	8	5
Differences	6	5	5	4	3	2	1

When this is accomplished the apparatus is lifted out of the cylinder D and the condenser detached. A light is then applied to the fuse, the condenser *quickly* replaced, and the whole plunged into the water; when gas appears through the holes at the bottom of A the time is noted, and an observation made as to the regularity of the combustion. When the combustion is at an end, which should in most cases occupy not less than 60 seconds, the stopcock of A is opened and a wire forced down the pipe to clear it; the whole is then moved up and down in the liquid with the thermometer until the temperature has attained its maximum, and the increase is then noted.

Often there is a small quantity of sample that has escaped combustion; and to make allowance for this, the liquid is made acid with hydrochloric acid, and evaporated to a small bulk in a porcelain basin. The residue is filtered off, washed, dried, brushed into a tared crucible, dried and weighed; it is then ignited in a muffle, cooled, and again weighed; the loss is assumed to be the carbon and hydrogen unburnt.

To ascertain the temperature that this would raise the water to if completely burnt in the calorimeter: Let V = combustible matter minus water in 2 grms. sample, C amount of carbon and hydrogen found to be unburnt, and T the rise in temperature in calorimeter, then the temperature T_2 corresponding to C will not be far short of,

$$\frac{T \times C}{V} = T_2$$

The following is an example of the determination of the heating power of a sample of Welsh steam coal by the above process:—

Temperature of room	17·25° C.
" " water before combustion .	13·20° C.
" " " after " . .	20·30° C.
Increase in temperature 20·3 — 13·2 . =	7·10° C.
Allowance for temperature of C and H unburnt	·21° C.
	7·31° C.
Absorption of heat by calorimeter $\frac{1}{10}$. =	·73° C.
Total	8·04° C.

8·04 × 1,000 = 8,040 calories—that is, pounds of water heated 1° C. by 1 pound of sample.

The latent heat of steam being 537 thermal units, the evaporative power (lbs. of water evaporated at 100° C. (212° F.) by 1 lb. of coal) becomes $\frac{8040}{537} = 14·97$.

On determining the heating power of substances rich in hydrogen—such as patent and liquid fuels—the oxygen mixture is diluted with from ·5 to 3 grams of dry kaolin clay. The combustion is sometimes difficult to start; in most cases, however, this may be overcome by the employment of a little gunpowder or coal mixture with the fuse, the calorific value of which has been previously ascertained; and the temperature corresponding to the amount taken, must be subtracted from the increase of temperature observed.

The following are details of the process adopted by the writer for the determination of the heating power of liquid fuels:—16 grams of dry oxygen mixture are mixed with 2 grams of recently ignited kaolin and spread out over a porcelain plate. A small beaker containing about three grams of the liquid, together with a small glass pipette, are accurately weighed; then 1·5 grams are taken off the weight-pan of the balance. The liquid is now sucked up into the pipette and dropped over the surface of the oxygen mixture and kaolin, until, on re-weighing, the balance is in equilibrium; 1·5 grams will thus have been taken for the test. It need not matter that exactly 1·5 grams be taken so long as what is taken is known, since the amount of water used in the calorimeter can be adjusted to meet the case. The mixture is now thoroughly mixed with the aid of a spatula and carefully transferred to the copper cylinder. A mixture of about ·04 gram of coal (the calorific value of which has been previously ascertained) and 0·5 gram of oxygen mixture is brushed on the top of the mixture in the cylinder, and a cotton fuse inserted. The rest of the process is conducted in the manner described, except that the quantity of water taken is 1,500 cc., or a quantity corresponding to 1 part of sample in 1,000 cc. of water. By this process the writer has obtained the following results:—

A sample of crude American petroleum gave an evaporative power of 16·24 lbs.

A sample of crude Russian kerosene gave an evaporative power of 16·75 lbs.

A sample of coal tar creosote gave an evaporative power of 13·65 lbs.

These results, however, must be considered slightly below the truth, since there is always a little evaporation going during the mixing of the sample, &c.

Calculation of the Theoretical Calorific Values of Solid and Liquid Fuels from the Chemical Analysis.—Carbon and hydrogen are the only elements in solid and liquid fuels that may be considered the source of their heating efficiency, consequently the amount of heat that would be expected from them would be thought to depend upon the respective amounts of these elements present in the fuel. The quantity of oxygen present, however, has to be taken into account, which is assumed to be combined with its equivalent of carbon or hydrogen as the case may be, and renders so much of the latter incapable of generating heat.

The amount of heat rendered ineffective by the quantity of oxygen present, will depend upon whether the latter is supposed to be combined with carbon, or hydrogen. The amount of carbon rendered latent by a given quantity of oxygen, would be three times as much as the amount of hydrogen rendered latent by the same quantity of oxygen.

The heat given out in the combustion of hydrogen is always the same, but in the case of carbon it depends upon whether it is oxidized to its maximum as carbonic acid (CO_2) or to its minimum carbonic oxide (CO), so that carbon can have two calorific values according whether the product of combustion be CO or CO_2.

In practical working, great loss of heat would be entailed if the carbon were not oxidized to its full, as the following shows:—

		Calories.
The heat generated by the combustion of carbon to carbonic acid is	=	8080
While the heat generated by the combustion of the same weight of carbon to carbonic oxide is . .	=	2473
Loss of heat by production of CO	=	5607

From accurate determinations made by Favre and Silbermann it has been found that the amount of heat generated by the combustion of hydrogen is 4·265 times as great as the heat given out by the combustion of the same weight of carbon to carbonic acid.

The relative calorific power of fuels may be deduced from the following formulæ:—

(1.) Fuel containing carbon only . . $p = C$.
(2.) Fuel containing carbon and hydrogen $p = C + 4{\cdot}265\,H$.
(3.) Fuel containing carbon, hydrogen, and oxygen . . . . $p = C + 4{\cdot}265\,(H - \frac{1}{8}O)$.

where, p = relative calorific power

C, H and O = amounts of carbon, hydrogen, and oxygen present in 1 part of fuel.

If it be required to express the calorific power of a fuel in heat units then—

(1.) $p = 8080\,C$.
(2.) $p = 8080\,C + 34462\,H$.
(3.) $p = 8080\,C + 34462\,(H - \frac{1}{8}O)$.

The following table gives the calorific values of several substances as calculated from the above formulæ :—

Fuel.	Composition of fuel.				Relative calorific power.	Heat Units, Centigrade.	Weight of Water heated from 0° to 100° C.	Wght. of Water at 100° C. converted into Steam.
	Carbon.	Hydrogen.	Oxygen.	Ash.				
Hydrogen . .	—	1·00	—	—	4·265	34,462	344·62	62·658
Marsh gas . .	0·75	0·25	—	—	1·816	14,675	146·75	26·682
Olefiant gas .	0·875	0·143	—	—	1·466	11,849	118·49	21·543
Welsh coal . .	0·838	0·048	0·041	0·049	1·020	8,241	82·41	14·983
Newcastle coal .	0·821	0·053	0·057	0·038	1·017	8,220	82·20	14·945
Carbon . .	1·000	—	—	—	1·000	8,080	80·80	14·691
Scotch coal .	0·785	0·056	0·097	0·040	0·973	7,861	78·61	14·292
Derbyshire coal .	0·797	0·049	0·101	0·026	0·956	7,733	77·33	14·060
Lancashire coal .	0·779	0·053	0·095	0·049	0·955	7,717	77·17	14·031
Kiln-dried peat .	0·600	0·060	0·307	0·020	0·694	5,640	56·40	10·254
Air-dried peat .	0·461	0·046	0·246	0·015	0·526	4,250	42·50	7·727

In determining the thermal effect of fuels, from their percentage composition, when made to burn in air, corrections have to be made for the latent heat of water, produced by the combustion of the available hydrogen ; and the specific heats of the carbonic acid, water vapour, nitrogen, and air, have also to be taken into account. The following is a formula for arriving at the thermal effect of a fuel when completely oxidized in air :—

$$T = \frac{cC + c'H - lW}{S\ 3{\cdot}67\ C + 9\ H + S'\ W + S''\ N + S'''\ A}.$$

Here T = increase of temperature produced by combustion.
C and H = quantities of carbon and hydrogen available in 1 part fuel.
W = water produced by 1 part fuel.
l = latent heat of water.
$S\ S'\ S''\ S'''$ = specific heats of carbonic acid, water vapour, nitrogen and air.
C and C' = calorific power of carbon and hydrogen.
N = nitrogen in quantity of air necessary for complete combustion of fuel.
A = any additional amount of air supplied for combustion.

The result obtained by the above formula expresses the highest heat attainable as compared with carbon burnt to its highest oxide under the best conditions. The amount of heat generated practically, however, is usually less than what should be obtained by calculation as above; this is due to a variety of causes, such as imperfect combustion, loss of fuel as smoke, imperfectly oxidized cinders, &c.

Rankine adopts as his unit, the weight of fuel required to evaporate 1 lb. of water at 212° F. (= 100° C.) under a pressure of 14·7 lbs. per square inch; this being equivalent to 966 British thermal units. The results were obtained as follows :—

Let E be the corrected and reduced evaporation,
e the weight of water evaporated,
T_1 the standard boiling point, 212° F. (= 100° C),
T_f the temperature of feed water,
T_b the actual boiling point observed;

then,

$$E = e \left\{ 1 + \frac{T_1 - T_f + 0{\cdot}3\,(T_b - T_1)}{966\ \text{F. or } 537\ \text{C.}} \right\}$$

The result is the number of times its own weight of water which a fuel would evaporate at the standard temperature, if no loss of heat occurs; but as there always is some loss of heat, the efficiency of the furnace is expressed by the ratio, $\frac{E\ (\text{Available})}{E\ \text{Total}}$, which, if no waste occurred, would be = 1.

The loss of units of evaporation by the waste gases may be obtained by the formula,

$$\text{Loss by chimney } \frac{1 + A'}{4000} \text{ Tc (F.°)}$$

Here $1 + A' =$ weight of burnt gas per unit weight of fuel, and Tc (F.°) the temperature of the chimney gases above that of the atmosphere. For ordinary coal $1 + A'$ ranges from 13 to 25, and for liquid fuels 16·3, if no excess of air is required.

Some examples with coal, with a chimney draught, the temperature of the waste gases being 600° F.(= 315° C.), were,

$1 + A'$	=	13	19	25
Tc	=	600°	600°	600°
Vol. of gases in cubic feet . .	=	325	475	625
Loss of evaporative power . .	=	1·95	2·85	3·75

In estimating the evaporative efficiency of fuels from their chemical constitution, Rankine proposed the formula,

$$E = 15C + 64H - 8O;$$

and to calculate the amount of air required for combustion, $A = 12\,C + 36\,H - 4\tfrac{1}{2}\,O$. The practical value of a fuel, however, is a little lower than that estimated by the formula. The following results were obtained by Rankine:—

	Chemical Composition.					Evaporation due to	
	C.	H.	O.	A.	E.	C.	$H-\frac{O}{8}$.
Charcoal . .	·93	0	0	11·5	14·0	14·0	0
Coke . . .	·88	0	0	10·6	13·2	13·2	0
Rock oils $C_{18}H_{20}$	·84	·16	0	15·75	22·7	12·7	10·0
Rock oils $C_{26}H_{28}$	·85	·15	0	15·65	22·5	12·66	9·84
Coal . . .	·87	·05	·04	12·1	15·9	13·05	2·85
" . . .	·85	·05	·06	11·7	15·5	12·75	2·75
" . . .	·75	·05	·05	10·6	14·1	11·25	2·85
Ethylene . .	·75	·25	0	18·0	27·3	11·25	16·05
Acetylene . .	·85	·14	0	15·43	22·1	12·9	9·2
Peat, dry . .	·56	·06	·31	7·7	10·0	8·5	1·5
Wood, dry . .	·58	·05	·40	6·0	7·5	7·5	0

Theoretical Value of Liquid Fuels.—Dr. Paul determines the evaporative power of hydrocarbons as the sum of that of the hydrogen and carbon present, assuming that when oxidized with the theoretical proportion of air, each lb. of carbon evaporates 11·359 lbs. of water at 15·5° C., and each lb. of hydrogen 41·895 lbs. of water at 15·5° F., into steam at 100° C. The results obtained by this method are given in the following table. Column 5 gives the evaporative duty when the furnace gases are discharged at 315° C. above the temperature of the air supplied to the furnace.

	Carbon.	Hydrogen.	Oxygen.	Evaporative Power, lbs. water at 100° C.	Evaporative Duty, lbs. water at 15·5° C.
Phenol	76·6	6·40	17·00	12·2437	10·5025
Cresol	77·77	7·41	14·82	13·0096	11·1632
Naphthalin	93·75	6·25	—	15·4350	13·0751
Anthracine	94·38	5·62	—	15·2417	13·2675
Xylol	90·56	9·44	—	16·5866	14·2415
Cumol	90·00	10·00	—	16·7838	14·4126
Cymol	89·55	10·45	—	16·9422	14·5500

It is computed, generally speaking, that in average practical working, 1 lb. of liquid fuel would not be likely to evaporate more than 16 lbs. of water as steam.

An example of Paul's method of obtaining the *effective* heat is as follows:—

COMBUSTION OF 1 LB. OF CARBON.

	Heat Units.	Equivalent Evaporation of Water.	
		At 212° F.	At 60° F.
Total heat of combustion	14,500	15	—
Available heat	14,500		—
Waste by furnace gases at 600° F.	3,480	3·6	—
Effective heat	11,020	11·4	9·8

Combustion of 1 lb. of Hydrogen.

	Heat Units.	Equivalent Evaporation of Water.	
		At 212° F.	At 60° F.
Total heat of combustion	62,032	64·2	—
Latent heat of water vapour . . .	8,695	—	—
Available heat	53,337	—	—
Waste heat of furnace gases . . .	11,520	11·9	—
Effective heat	41,817	42·3	38

The following are the results obtained per lb. of two kinds of liquid fuel, A and B:—

A containing 86 per cent. carbon and 14 per cent. hydrogen.
B „ 75 „ „ 25 „ „

A.

Carbon.	Hydrogen.		Total Heat of Combustion.	Equivalent Evaporation of Water.	
				At 212° F.	At 60° F.
·86		× 14500 =	12470		
	·14	× 62032 =	6684		
			21154	21·9	18·8
Furnace Gases.			Heat units in Furnace Gases.		
		lbs.			
Carbonic acid . .		3·16	411		
Water vapour . .		1·26	359		
Nitrogen . . .		11·45	1683		
Surplus air . .		14·37	2124	2·2	
		30·74	4577	4·8	
Total heat of combustion . . .			21154		
Latent heat of water vapour . .			1217	1·3	
Available heat			19937		
Waste in furnace gases . . .			4577	4·8	
Effective heat			15360	15·8	13·6
Theoretical evaporating power				21·9	

B.

Carbon.	Hydrogen.		Total Heat of Combustion.	Equivalent Evaporation of Water.	
				At 212° F.	At 60° F.
·75		× 14500 =	10775		
	·25	× 62032 =	15508		
			26283	27·1	23·1
Furnace Gases.		lbs.	Heat units in Furnace Gases.		
Carbonic acid		2·75	358		
Water vapour		2·25	641		
Nitrogen		13·39	1968		
Surplus air		17·39	2483	2·6	
		35·78	5450		
Total heat of combustion			26283		
Latent heat of water vapour			2174	2·2	
Available heat			24109		
Waste in furnace gases			5450	5·6	
Effective heat			18659	19·3	16·6
Theoretical evaporating power				27·1	

Holden's System of Using Liquid Fuel on Locomotive and Stationary Boilers.—By this system liquid fuel and air are introduced into the fire-box above a thin layer of solid incandescent fuel by means of a special injector, Fig. 38, and burnt in combination with the solid fuel, without any alteration of the fire-box, other than the insertion of one or more tubes through the casings, the boiler so fitted being equally suitable for the use of ordinary fuel. The use of this method results in effectual combustion, entire absence of smoke, intense and regular heat, and great economy of fuel. Slack, inferior coal, lignite, cinders, wood, peat, or sawdust may, with equally good results, be used as solid fuel. The air necessary for combustion, not having to be introduced through the fire, a very small amount of draught is required, and in the case of locomotives, the orifice of the blast pipe may advan-

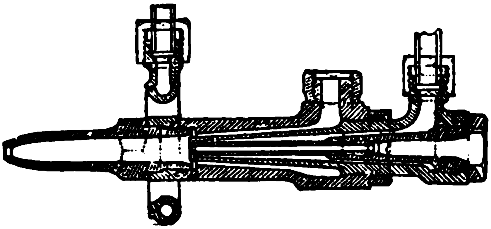


FIG. 37.—STATIONARY PATTERN.

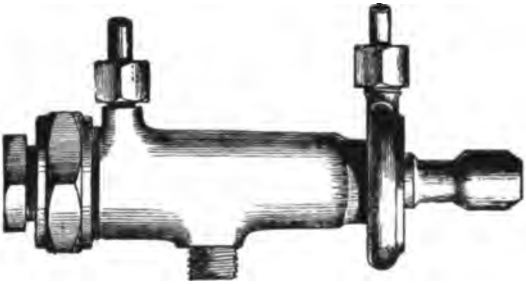


FIG. 38.—HOLDENS' LIQUID-FUEL INJECTOR.

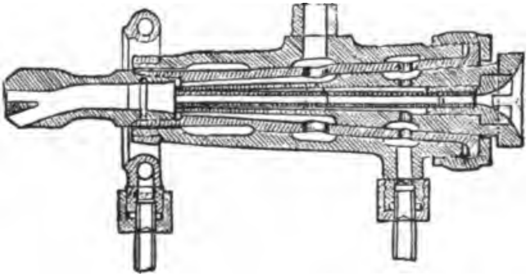


FIG. 39.—LOCOMOTIVE PATTERN.

tageously be enlarged from 50 to 60 per cent., thus reducing the wear and tear of the fire-box, tubes, smoke-box, and chimney, preventing the emission of sparks and ashes, and conducing to economic and efficient working by the diminution of back pressure.

The steam pressure in a boiler fitted with the apparatus can be regulated with great nicety, as the solid fuel required is only sufficient to form a base for ignition, and the steam pressure can be promptly increased or reduced by varying the quantity of liquid fuel. For locomotives this is specially advantageous, as, in the case of an exceptionally heavy load, strong wind, adverse gradient, or an engine employed piloting or shunting, a good supply of steam can be quickly ensured and easily maintained. On the other hand, if an engine is suddenly stopped by signals, or has to put by for other trains to pass, the generation of steam can be immediately stopped.

In the event of tubes or stays leaking, or an engine having to face a bank towards the end of a journey when the fire is low, steam can be maintained and the train worked with the help of liquid fuel, when without it it would probably lose time and come to a stand.

It is therefore submitted that even in cases where the relative cost of coal and liquid fuel is in favour of coal burning, the advantages to be derived from the system are sufficient to render its adoption desirable as a supplementary means of meeting any exceptional demands upon the engine. For results obtained by this system see page 185.

GASEOUS FUELS.

The great progress that has recently been made in the manufacture and application of gas suitable for fuel, metallurgical and domestic heating purposes, &c., necessitates a ready method by which its analysis and heating power can be quickly deduced with fair practical accuracy.

For the very accurate and scientific analysis of complex gas mixtures, delicate processes, such as Frankland and Ward's, would have to be resorted to, which would, generally speaking, be far too tedious and elaborate for practical working purposes. In metallurgical works, water-gas works, &c., it is often necessary to have several complete technical analyses of gas in a single day, in order to give an idea of the economic working of processes, &c.

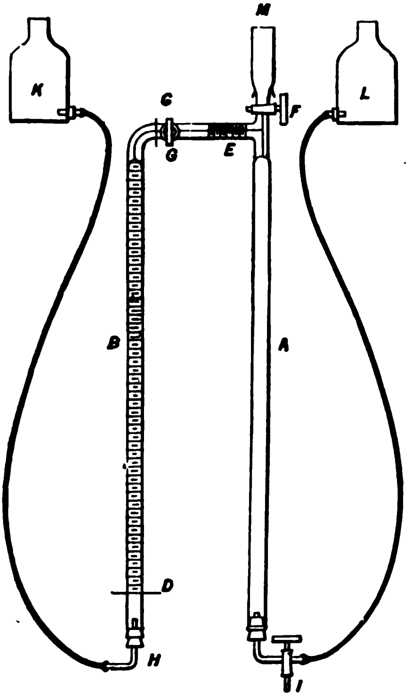


Fig. 40.

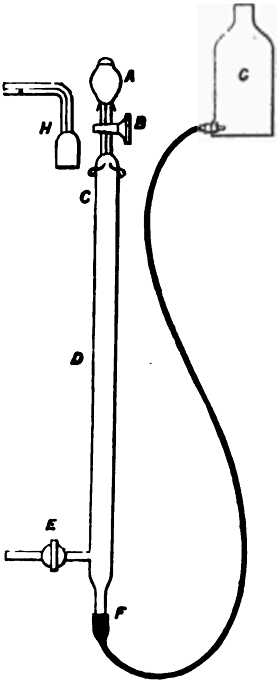


Fig. 41.

The apparatus which seems to commend itself for quick working and reasonable accuracy is that devised by Elliot, of which a sketch is here given. B, Fig. 40, is a tube graduated to

100 cc. in $\frac{1}{10}$th cc. The stop-cock I is a three-way tube with a delivery tube through its stem. The bottles K and L hold about a pint each. M is a portable funnel ground to fit above F and holds 60 cc. E is a rubber tube joining A and B.

Before starting an analysis, the tubes A and B are filled with water from the bottles K and L, by the manipulation of the stop-cocks C, F, and I; when water rises into the funnel M, and all air eliminated, F and G are turned off, the funnel M removed, and the tube containing the gas for analysis attached in its place; the bottle L is now slowly lowered and stop F closed; remove the gas tube from F, replace the funnel M, raise the bottle L, lower the bottle K, and open the stop-cock G. The gas is thus driven into the graduated tube B. Lower the bottle K so that the level of the water therein is in a line with the zero mark D. The gas is then adjusted to the zero mark D by the bottle L, the stop-cock G closed, and the temperature and pressure recorded.

When the surplus gas has been displaced from the tube A by raising L and opening F, by manipulating the bottles and stop-cocks the gas is now drawn into A. Close the cocks, lower the bottle L, and fill the funnel M with a 10 E solution of potassic hydrate. Cautiously open F and allow the liquid to flow into the tube A, always leaving, however, about 10 cc. in M. On allowing to stand until no further diminution in volume occurs, due to the absorption of carbonic acid (CO_2) present in the gas, the residual gas is transferred to the tube B and measured, the temperature and pressure being noted; the loss on the original volume is CO_2. Empty the tube A, wash out with water, and refill with water as before. The gas from B is transferred to it, the funnel M is half filled with water, and a few drops of bromine added; this is run into the tube until fumes of bromine are seen to be mixed with the gas.

On allowing to stand until no further contraction occurs, due to the absorption of ethylene and other illuminants, some of the potash used for the absorption of the CO_2 is added; this absorbs the excess of bromine. When this is complete the gas is measured as before, the loss in volume being put down to illuminants.

After A has been cleared out and refilled with water, the gas is again brought into it as before; the funnel M is filled with a solution of 8 E potassic hydrate, to which has been added about 3 per cent. of pyrogallic acid; this is run into the tube and the gas allowed to remain over it, until any oxygen present is completely absorbed; it is then measured, loss = oxygen. Clean out A, withdraw the gas from B into it, fill M with 10 E hydrochloric acid, containing 25 per cent. of cuprous chloride, and allow to stand till no further diminution in volume occurs, and measure as before; the loss is due to carbonic oxide (CO).

The residual gas may now contain marsh gas, hydrogen, and nitrogen; and to determine the proportion of these it will be necessary to explode them with oxygen by an electric spark. This is accomplished by means of the explosion tube, Fig. 41, graduated to 100 cc. in $\frac{1}{10}$ cc. to within 2 inches from E, the stop-cock B being the zero point. The funnel A is portable as M in Fig. 40; at C there are two platinum wires fused in, which are connected with an induction coil. The bent tube H is made to fit over the stop-cock B when the funnel A is removed, which serves to facilitate the transfer of gas. Before starting, remove the funnel M in the absorption tube, and fix in its place a bent tube like H. The gas having been transferred to A, the explosion tube is placed near it, and the bent tube H is attached to a piece of rubber tube, long enough to reach to the corresponding bent tube of the explosion tube.

The explosion tube is now filled with water from the bottle G, to the end of the tube H over the stop-cock B. Fill the piece of tubing with water, and connect the two bent tubes H with it. Now turn the three-way cock I, so that the bottom of the tube A is closed. Now open B and F, and by the aid of the bottles draw in 20 cc. of the gas into the explosion tube, manipulating with the bottle G, and close the stop-cocks. Connect the inlet tube E with a gas-holder of oxygen under pressure, and introduce about 20 cc. into the tube, and mix; level off and note volume. Place the bottle G below F to expand the gases, pass the spark, and with a click the explosion is complete. Allow the flush of heat to debate, and observe the contraction after levelling with G.

By removing the bent tube H and fixing on the funnel A, the amount of CO_2 produced can be ascertained by introducing the solution of potassic hydrate, and observing the diminution in volume. The formula for the calculation of the proportion of hydrogen, marsh gas, and nitrogen present in the 20 cc. of residual gas taken (which must afterwards be calculated on the original gas taken), becomes :—

Let A = original vo'ume;
C = contraction;
D = carbonic acid formed;
X, Y, Z = H, CH_4, and N respectively;

then—

$$X = \frac{2C - 4D}{3}$$
$$Y = D$$
$$Z = \frac{3A - 2C + D}{3}$$

In working with the apparatus, the analysis should be performed in a room where the temperature would remain uniform during the analysis, and care should be taken that the water, chemicals, &c., are at the same temperature as the room.

Inasmuch as a complete analysis can be made by this process in the course of an hour, the temperature and pressure, if suitable precautions be taken, would rarely be altered during the readings; and the original gas being usually saturated with moisture, no correction would be necessary for the tension of aqueous vapour, since it is measured over water.

Estimation of Sulphur in Gaseous Fuel.—It is quite as important that gases intended for heating purposes should contain the least possible quantity of sulphur as coals, cokes, &c. The condition in which the sulphur exists in gaseous fuels is of secondary importance, since the ultimate product of combustion is usually the same, viz., *Sulphurous Anhydride* (SO_2); so that it will be necessary to estimate total sulphur only, and not to determine the amounts of sulphuretted hydrogen, bisulphide of carbon, &c., separately. The best practical method the writer is acquainted with for the determination of the total sulphur is that devised by the late Dr. Letheby, the principle of which is that a known volume of gas is burnt in an atmosphere of air and ammonia. The sulphurous anhydride thus produced is neutralized by the ammonia, forming sulphite and sulphate of ammonia, which is condensed, dissolved in water, the sulphite oxidized to sulphate by chlorine water, and the sulphur precipitated by Baric chloride, and estimated in the usual manner. This process is the one adopted by the Metropolitan Gas Referees for the determination of sulphur other than sulphuretted hydrogen in illuminating gas. In this case the sulphuretted hydrogen is absorbed by acetate of lead papers, and any ammonia that is present is absorbed by sulphuric acid contained in a tube filled with glass beads previous to the process about to be described.

Dr. Letheby's Process for the Determination of Total Sulphur.—The apparatus employed for this method is represented in Fig. 42. It consists of the meter A, by which the volume of gas taken for the estimation is ascertained; B a Bunsen burner with a steatite top, with a receptacle for pieces of sesquicarbonate of ammonium. C is a condensing funnel or trumpet-tube, and connected with the cylinder D filled with glass beads, and which serves to complete the condensation of the ammonium sulphate, &c., produced. In the bottom of the cylinder is fixed a small glass tube through which the liquid (formed during the testing) drops into the beaker G, placed underneath.

When all is fixed ready for testing, as in sketch, and small pieces of pure sesquicarbonate of ammonium, weighing in all about two ounces, have been placed between the narrow stem of the burner and the trumpet-tube, the gas is made to flow through the meter at the rate of half a cubic foot per hour, and is lit at the Bunsen burner. When 10 cubic feet have passed through, the gas is turned off, and the average temperature and pressure observed. The cylinder D and the tube C are then washed out with distilled water into the beaker G. To the total liquid is now added a little chlorine water to oxidize any sulphite present into the state of sulphate. The liquid is now boiled, acidulated with pure hydrochloric acid, and the sulphur precipitated with Baric chloride and estimated as described on page 125. Of course it will be necessary to correct the observed 10 cubic feet of gas to the standard temperature and pressure, viz., 60° F. (15·5° C.) and 30″ or 760 mm. of mercury (*vide* page 129).

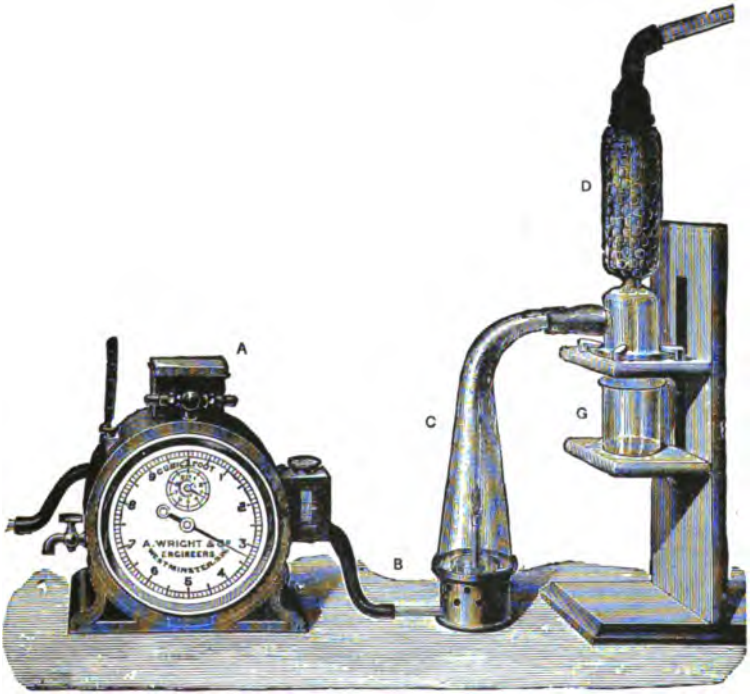


FIG. 42.

It may here be added that the maximum amount of sulphur other than sulphuretted hydrogen allowed by the Gas Referees is 22 grains of sulphur in every 100 cubic feet of gas.

Calorific Value of Gaseous Fuels.—The absolute heating effect (A) of a gaseous fuel containing carbonic acid, hydrogen, marsh gas, olefiant gas, nitrogen, carbonic acid, and aqueous vapour, Bunsen deduces by the following formula. Granting that one part of carbonic oxide combines with 0·57 parts of oxygen; 1 part of hydrogen with 8 of oxygen; 1 part of marsh gas with 4 of oxygen; 1 part of olefiant gas with 3·43 parts oxygen; and that the combustion of 1 part marsh gas forms 2·25 parts of water; and 1 part of olefiant gas 1·29 parts of water:—

$$A = 3000\,[x \cdot k \cdot o \cdot 57 + 1{\cdot}5 \cdot h \cdot 8 + 1{\cdot}1 \cdot g \cdot 4 + 1{\cdot}17 \cdot o \cdot 3{\cdot}43] \\ - 550\,[g \cdot h \cdot + 2{\cdot}25 \cdot g + 1{\cdot}29 \cdot o + w]$$

Where K = amount of Carbonic Oxide (CO)
$h =$ " Hydrogen (H)
$g =$ " Marsh gas (CH_4)
$o =$ " Olefiant gas (C_2H_4)
$n =$ " Nitrogen (N)
$w =$ " Aqueous vapour (OH)
$k =$ " Carbonic acid (CO_2)

The co-efficient of intensity for carbonic oxide is expressed by x, as it remains doubtful whether it is 1·44 or 1·1.

For the pyrometric heating effects of gaseous fuel burnt in air the following formula is used. Assuming that 1 part of carbonic oxide is converted into 1·57 parts of carbonic acid; 1 part of marsh gas into 2·75 parts of carbonic acid and 2·25 of water; and 1 part of olefiant gas gives 3·14 parts of carbonic acid and 1·29 parts of water:—

$$T = \frac{A}{Q(K)S + Q(w)S' + Q(n)S''}$$

$$Q(K) = K + 1{\cdot}57 \cdot k + 2{\cdot}75 \cdot g \cdot + 3{\cdot}14 \cdot o$$

$$Q(w) = w + 9 \cdot h + 2{\cdot}25 \cdot g + 1{\cdot}29 \cdot o$$

$$Q(n) = n + 3{\cdot}33\,(o \cdot 57 \cdot k + 8 \cdot h + 4 \cdot g + 3{\cdot}43 \cdot o)$$

$SS'S''$ = specific heats of CO_2, OH_2, and N respectively. A much higher temperature would be produced if burnt in pure oxygen, as the following calculation by the above formula shows:—

	In oxygen.	In air.
Carbon	9873° C.	2458° C.
Carbonic oxide	7067	3042
Olefiant gas	9187	5413
Marsh gas	7857	5329
Hydrogen	8061	3259

As a general rule the amount of heat generated by a compound substance is less than that which is given out by its individual constituents on combustion, although not uniformly so; and it appears that polymeric bodies (substances which have the same percentage composition but which contain a different number of atoms in their molecule) give out different amounts of heat. The following table indicates the amount of heat given out by the Hydrocarbons polymeric with olefiant gas.

		Heat Units.
Olefiant gas	$C_2 H_4$	11858
Amylene	$C_5 H_{10}$	11491
Paramylene	$C_{10} H_{20}$	11303
Cetene	$C_{16} H_{32}$	11055
Metamylene	$C_{20} H_{40}$	10928

METHOD OF ASCERTAINING THE THERMIC VALUE OF A GAS COMPARED WITH COAL.

(*Ford, Jour. I. and S. Inst.*)

A natural gas from the Pittsburg district has the following average chemical composition :—

Carbonic acid	0·60	per cent.
Carbonic oxide	0·60	,,
Oxygen	0·80	,,
Olefiant gas	1·00	,,
Ethylic hydride	5·00	,,
Marsh gas	67·00	,,
Hydrogen	22·00	,,
Nitrogen	3·00	,,

Now by the specific gravity of these gases we find that 100 litres of this gas will weigh 64·8585 grms., thus :—

Marsh gas .	67·0	litres	weighs	48·0256	grms.
Olefiant gas .	1·0	,,	,,	1·2534	,,
Ethylic hydride .	5·0	,,	,,	6·7200	,,
Hydrogen . .	22·0	,,	,,	1·9712	,,
Nitrogen . .	3·0	,,	,,	3·7632	,,
Carbonic acid .	·6	,,	,,	1·2257	,,
Carbonic oxide .	·6	,,	,,	0·7526	,,
Oxygen . .	·8	,,	,,	1·1468	,,
Total				64·8585	,,

Then if we take the heat units of these gases, we will find

Marsh gas .	48·0256	grms. contain	627,358	heat units.
Olefiant gas .	1·2534	,,	14,910	,,
Ethylic hydride	6·7200	,,	77,679	,,
Hydrogen .	1·9712	,,	67,929	,,
Carbonic oxide	·7526	,,	1,808	,,
Nitrogen .	3·7630	,,	—	,,
Carbonic acid .	1·2257	,,	—	,,
Oxygen . .	1·1468	,,	—	,,
	64·8585	,,	789,694	,,

64·858 grams are almost exactly 1,000 grains, and 1 cubic ft. of this gas will weigh 267·9 grains; then the 100 litres or 64·8585 grams or 1,000 grains are 3·761 cubic ft.

3·761 cubic ft. of this gas contain 709,694 heat units, and 1,000 cubic ft. will contain 210,069,604 heat units. Now 1,000 cubic ft. of this gas will weigh 265,887 grains, or 38 lbs. avoirdupois.

We find that 64·8585 grms. or 1,000 grains of carbon contain 524,046 heat units, and 265,887 grains or 38 lbs. of carbon contain 139,398,896 heat units. Then 57·25 lbs. of carbon contain the same number of heat units as 1,000 cubic ft. of the natural gas, viz., 210,069,604.

Now, if we say that coke contains in round numbers 90 per cent. of carbon, then we will have 62·97 lbs. of coke equal in heat units to 1,000 cubic ft. of natural gas.

Then, if a ton of coke, or 2,000 lbs., cost 10s., 62·97 lbs.

will cost 4d., or 1,000 cubic ft. of gas is worth 4d. for its heating power.

We will now compare the heating power of this gas with bituminous coal, taking as a basis a coal slightly above the general average of the Pittsburg coal, viz.—

Carbon	82·75 per cent.
Hydrogen	5·31 ,,
Nitrogen	1·04 ,,
Oxygen	4·64 ,,
Ash	5·31 ,,
Sulphur	0·95 ,,

We find that 38 lbs. of this coal contain 146,903,820 heat units. Then 54·4 lbs. of this coal contain 212,069,640 heat units, or 54·4 lbs. of coal are equal in its heating power to 1,000 cubic ft. of natural gas. If our coal costs us 5s. per ton of 2,000 lbs., then 54·4 lbs. will cost 1·632 pence, and 1,000 cübic ft. of gas will be worth for its heat units 1·632 pence.

As the price of coal increases or decreases, the value of the gas will naturally vary in like proportions. Thus with the price of coal at 10s. per ton, the gas will be worth 3·264 pence per 1,000 cubic ft.

If 54·4 lbs. of coal be equal to 1,000 cubic ft. of gas, then 1 ton, or 2,000 lbs., will be equal to 36,764 cubic ft. or 2,240 lbs. of coal will be equal to 40,768 cubic ft. of natural gas.

If we compare this gas with anthracite coal, we find that 1,000 cubic ft. of gas are equal to 58·4 lbs. of this coal, and 2,000 lbs. of coal are equal to 34,246 cubic ft. of natural gas. Then if this coal costs 26s. per ton, 1,000 cubic ft. of natural gas will be worth 9½d. for its heating power.

Hartley's Calorimeter for Obtaining the Practical Calorific Value of Gaseous Fuels.—A practical method which will at once convey an idea of the amount of heat that a given gas will produce on combustion has been invented by Hartley, and gives results which, for a consumer, would be found as accurate as need be desired. In this process a given volume of gas is gradually burnt completely, and the heat given out is imparted to a known volume of water at an observed temperature, and from the increase in temperature produced, the heating power may be calculated.

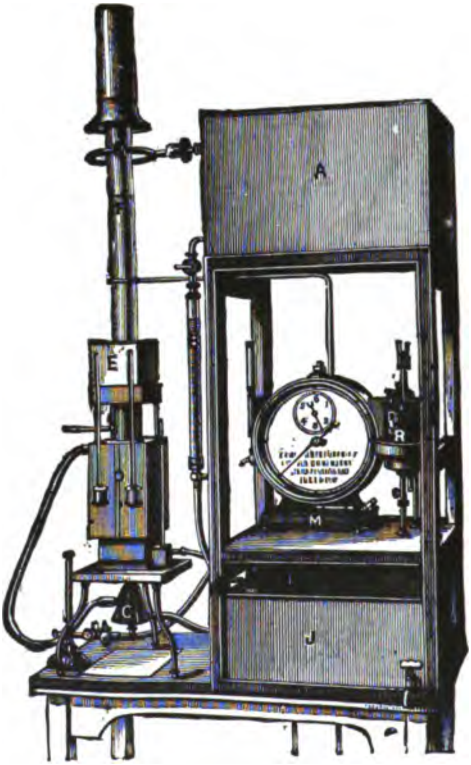


Fig. 43.

Fig. 43 is a sketch of the apparatus made by Messrs. Alex. Wright & Co., Westminster.

A is a small cistern from which the water it contains flows over a delicate thermometer in the tube B, and from thence through tubing into a case surrounding the stem of a special burner C, after which it traverses a jacket surrounding the calorimeter D, then makes its way to the top of D, and afterwards descends through a series of shelves exposing a very large surface, and eventually flows into the receiving tank J.

The gas is measured by the meter M, and its rate of consumption and the rate of the water flow are regulated until it be observed that the outflowing water is a few degrees higher in temperature than when it enters, and the temperatures indicated by the four thermometers used are found to be steady. The water during these adjustments is made to run away by a bye-way cock.

When all is ready for an experiment and the meter index at zero, the bye-way cock is turned off and the water made to flow into the receiving tank.

The quantity of gas usually burnt in each test is ¼ cubic foot, and occupies from 10 to 12 minutes.

When the ¼ cubic foot of gas has been burnt the water is turned off, and the increase in temperature of the water in the receiver J observed, and its weight taken. The weight of water multiplied by the increase in temperature gives the heating value of a ¼ cubic foot of gas.

Thus if 21 lbs. of water have been raised 9° F. by ¼ cubic foot of gas, then $21 \times 9 \times 4 = 756^\circ$ F. units for 1 cubic foot of gas. Of course the temperature and pressure correction must be made, and also loss of heat by conduction in the receiving tank, &c., added to the increase in temperature. This figure is determined once for all by observing the loss of heat that takes place when a known weight of water at a temperature of several degrees above that of the atmosphere is placed in the tank.

Specific Gravity of Gases.—The specific gravity of a gas used for heating purposes, &c., is of some value, since by its means an idea can be obtained of its economic production. The higher the temperature used to decompose coal, shale oil, &c., the lower will the density of the resulting gases be, owing to the decomposition of the heavier hydrocarbons with the deposit of carbon and formation of hydrogen and light hydrocarbons.

Dr. Letheby's Method of Determining the Specific Gravity.—The apparatus used for this method is shown in Fig. 44. It consists of a glass globe about 6 inches in diameter, furnished with two stop-cocks, to one of which is attached a tube 7 inches long and ½ inch in diameter, into which is fitted a thermometer and a jet for burning the gas. The other stop-cock is to be attached to a nozzle of the gas supply, and the gas allowed to flow through the apparatus and burnt at the jet until all air is expelled. When this is accomplished the supply cock is first turned off and then the upper one immediately afterwards, and the temperature and atmospheric pressure observed. The weight of the globe when filled with dry air at 15·5° C. (= 60° F.) and 760 mm. pressure is engraved upon it and a counterpoise weight is provided exactly equal to the weight of the globe when exhausted.

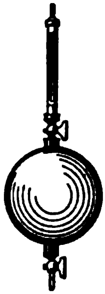


Fig. 44.

The globe is placed on a delicate balance, and the weight required in addition to the counterpoise noted.

Suppose that the weight required is ·995 gram and that the gas was dry, and the temperature was 60° F. and pressure 760 mm., and that the globe holds 2·285 grams of dry air at this temperature and pressure, then the specific gravity of the gas would be:—

$$\frac{0{\cdot}995}{2{\cdot}285} = {\cdot}435$$

But it is very rarely that the gas is weighed at the same temperature and pressure as the original air, and moreover the gas is usually moist. Corrections are, therefore, necessary to bring it under the same conditions. The following is an example how these corrections are made:—

We shall assume that the globe holds 2·285 gms. = 1867·54 cc. of dry air at 15·5° C. and 760 mm.

The weight of the moist gas collected at a temperature of 17° C. and a pressure of 755 mm. was 1·050 grams.

It is first necessary to reduce the volume of gas to 15·5° C. and 760 mm.

Now all gases increase practically $\frac{1}{273}$ part of their volume through each ° C. heated, and the volume of a gas is inversely as the pressure upon it. The tension of aqueous vapour increases with the temperature. The tension of aqueous vapour at a temperature of 17° C. is equal to a pressure of 14·42 mm. of mercury (see table, page 386), and in calculating the normal volume this figure must be subtracted from the observed atmospheric pressure, so that, applying the above principles, the volume of gas at 15·5° C. and 760 mm. becomes :—

$$V = \frac{1867{\cdot}5 \times (15{\cdot}5 + 273) \times (755 - 14{\cdot}42)}{(17 + 273) \times 760}$$

$= 1810{\cdot}41$ cc.

Now 1785·786 cc. of the gas at 15·5° C. and 760 mm. weigh 1·050 grams, and the globe has a capacity of 1867·543 cc.

Then the specific gravity of the gas at 15·5° C. and 760 mm. becomes :—

$$\frac{1867{\cdot}54 \times 1{\cdot}05}{1810{\cdot}41 \times 2{\cdot}285} = 0{\cdot}474$$

The Lux Specific Gravity Balance.*—An apparatus invented by Mr. Friedrich Lux for the automatic determination of the specific gravity of gases is now coming much into use, especially on the Continent. Fig. 45 is a sketch of the apparatus. It will be seen that it is constructed on the principle of the common lever balance, with a curved scale C, attached. The scale is graduated from 0 to 1, and the pointer D, moving in close proximity to it, enables the direct observation of the gas under examination to be made. The globe A holds 2 litres and is connected to the gas supply by the tube F, the gas thus passing through the tube G into the globe and hence through the tube B, where it may be burnt. The radiating heat from the flame, burning gas at the rate of 2 cubic feet per hour, exercises no appreciable difference on the gas in the globe.

---

* Can be obtained from Alex. Wright & Co., Millbank Street, Westminster, S.W.

The adjustment of the gas balance is made at 15° C., and 760 mm. pressure.

Before commencing the experiment the pointer D is adjusted, by means of the counterpoise E, until it rests at 1 on the scale. The gas is now turned on and allowed to flow through the globe for about ten minutes, when all air will be found to have been displaced. The exact reading is then taken, together with the temperature and pressure.

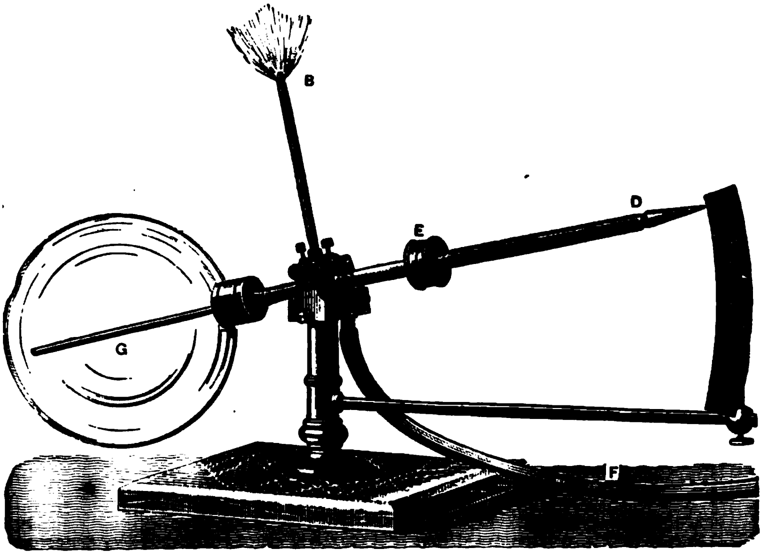


FIG. 45.

In very accurate determinations it must be remembered that in calculating the true specific gravity of the gas, the gas in the balance and the surrounding atmosphere becomes compressed by the increase of atmospheric pressure. Consequently, since the weight of the air is greater than that of ordinary lighting gas, it follows with an increase of pressure, one side of the balance becomes lighter to the amount of the difference, and *vice versâ* as the pressure decreases. In calculating the gravity of ordinary gas with a specific weight between 0·400 and 0·500, it will be found sufficiently accurate for practical purposes to add or deduct for every millimeter of pressure above or below 760 so many times the equivalent ·0007 ; and as many degrees as the temperature at the time of observation is more or less than 15° C., must be multiplied by ·002 and deducted or added as the case may be.

The following is an example :—

Temperature of gas, 30° C.
Pressure, 788 mm. Observed gravity 0·4350
30 − 15 = 15 ·002 × 15 = ·0300
788 − 760 = 28 ·0007 × 28 = ·0196
·0104 0·0104
Real sp. gr. = ·4246

On examining crude gases, which may contain liquid hydrocarbon, tar, &c., in suspension, it would be well to pass the gas through a tube containing cotton wool previous to its admission into the globe.

Chimney Draught.—To obtain an effective draught in a given chimney there should be a certain temperature of cold air used for combustion ; this can be obtained by calculation, and it has been found that the best chimney draught is obtained when the ratio of the temperature of the chimney gases to the external air is as 25 to 12. So it will be seen that this necessary heat required for the production of suitable draught means a great reduction in the intrinsic evaporative value of fuel.

On locomotives, where the size of the chimney does not admit of effectual combustion by the draught brought about by the hot gases alone, the required draught is produced by the steam blast-pipe : but owing to the rapid combustion and consequent escape of the hot gases at such high velocity there is no economy of heat by such a process of forced draught.

Rankine, on the question of chimney draught ("Steam Engine," p. 286), gives valuable formulæ on the basis of *absolute temperatures.* The following six formulæ will, no doubt, be often found valuable:—

Assuming that the volume of gas (or waste gases) from a furnace at 32° F. (= 0° C.) is 12½ cubic feet for each lb. of air supplied to the furnace, the volume of these gases at any other temperature T is—

$$V = \text{volume at } 32^\circ \times \frac{T + 461^\circ{\cdot}2}{493^\circ{\cdot}2} = V_0 . \frac{\tau}{\tau_0}$$

If—

w denotes the weight of fuel burned in a given furnace *per second;*
V_0 the volume at 32° of the air supplied per lb. of fuel;
τ_1 the absolute temperature of the gas discharged by the chimney;
A the sectional area of the chimney;
τ_0 being the absolute temperature of melting ice in degrees F.

Then the velocity of the current in the chimney in feet per second is

$$u = \frac{w V_0 \tau_1}{A \tau_0} \qquad (2)$$

And the density of that current in the chimney, in lbs. to the cubic foot, is very nearly

$$D = \frac{\tau_0}{\tau_1}\left(0{\cdot}0807 + \frac{1}{V_0}\right) \qquad (3)$$

The "head" required to produce this draught is according to a formula by Péclet, given by the equation,—

$$h = \frac{u_2}{2g}\left(1 + G + \frac{f l}{m}\right) \qquad (4)$$

Where—

l denotes the whole length of the chimney and of the flue leading to it in feet;

m denotes its "hydraulic mean depth," that is, its area divided by its perimeter, which for a square or round flue and chimney is one quarter of the diameter;

f is a co-efficient of friction, whose value for currents of gas moving over sooty surfaces is estimated by Péclet at 0·102; and

G is a factor of resistance for the passage of the air through the grate and the layer of fuel above it, whose value—according to Péclet when experimenting on furnaces burning from 20 to 24 lbs. of coal per square foot of grate—is 12.

Applying these values to the constants, then equation (4) becomes

$$h = \frac{u^2}{2g}\left(13 + \frac{0{\cdot}012\,l}{m}\right) \qquad (4a)$$

It appears that in using this formula, a conical or pyramidal chimney may, without sensible error, be treated as if it were cylindrical or prismatic with a uniform sectional area equal to that of the opening at the top. The same formula enables the velocity u to be computed when the head h is given. The head h is expressed in feet in height of a column of the hot gas in the chimney. It may be converted into an equivalent pressure in pounds on the square foot by multiplying by the density as found by equation (3) thus:—

$$\mathrm{p} = h\,\mathrm{D} \qquad (5)$$

And to express the head in inches of water the multiplier 0·192 is used: thus:—

$$\text{Head in inches of water} = 0{\cdot}192\,\mathrm{p} = 0{\cdot}192\,h\,\mathrm{D} \qquad (6)$$

Variable Blast Pipes on Locomotives.—A valuable appliance for economising fuel on locomotives is the somewhat recent invention by Mr. Macallan, M.I.M.E., of a variable blast pipe. The appliance is fully under the control of the driver, who can at will regulate an appropriate influx of air by increasing or decreasing, as the case may demand, the diameter of the nozzle of the blast pipe, by means of the adjustable cap A, shown in Fig. 46, which is manipulated from the foot-plate by a rod connected with the lever B. It is claimed that on an average a saving of 10 per cent. of fuel is effected by its means. The reason of this is not far to seek. The ordinary blast pipe of locomotives has an area such as will cause a sufficiently strong blast to enable the engine to make steam to do heaviest work, while when doing its light work the exhaust steam passes through the same orifice and consequently causes more fuel to be burnt than is required to raise the required amount of steam.

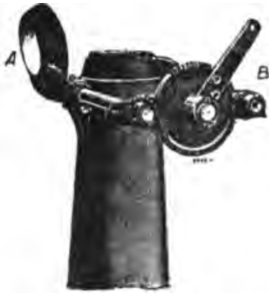


FIG. 46.—MACALLAN'S VARIABLE BLAST PIPE.

One great advantage of the above form of blast pipe over the ordinary kinds is that the frequent use of it prevents the formation of the sooty deposit usually seen in the latter, which causes a diminution of the orifice and entailing loss of fuel consequent upon the excess of blast produced.

Methods for the Analysis of Ashes of Fuels.—It is often desirable to know the composition of the ashes of fuel, since they play a somewhat important part in metallurgical operations, &c. The following process will be found to include the usual substances present in fuel ashes :—

Ignite in small quantities at a time about 200 to 300 grams of the sample in a shallow dish (platinum or porcelain), at a bright red heat in a muffle furnace; cool and put the ash in a stoppered bottle.

Determination of SiO_2, Fe_2O_3, CaO, MgO, *and* SO_4.—Weigh out 2 grams of the ash into a platinum crucible of about 50 cc. capacity, and mix with 8 grams of Fresenius' flux ($Na_3\ CO_2 + K_2\ CO_3$) ; place the lid on, and keep in a steady state of fusion over a blow-lamp for about a quarter of an hour. Cool ; place the crucible and its contents into a beaker of about 250 cc.

capacity, and pour on to it 50 cc. of pure dilute hydrochloric acid (25 cc. HCL; sp. gr. 1·16 + 25 cc. of water). Place cover on beaker and gently warm until all that is soluble is dissolved; raise crucible out of solution with the aid of a pair of bone forceps or a stir-rod, and detach any adhering silica with the aid of a feather and a jet of water from a wash bottle. The solution is now evaporated to dryness over a water bath and the residue heated on a sand bath for about an hour at a temperature of about 130° C., to render silica insoluble, after which it is cooled, the residue moistened with 10 cc. of 10 E hydrochloric acid, and gently warmed for about ten minutes over a water bath. 200 cc. of water are now added, and the solution boiled until all that is soluble is dissolved. The insoluble residue is now pure silica, the bases being in solution as chlorides. Filter off the silica and wash with a boiling solution of dilute hydrochloric acid (1 in 20) until the washings no longer produce a red coloration, when a little is mixed with a few drops of potassic sulphocyanate solution on a watch-glass. Reserve the solution. Detach the filter and its contents from the funnel, spread out on a watch-glass, and dry in a water-oven; in the meantime, accurately weigh a platinum or porcelain crucible of convenient size. After the silica has dried, transfer it to the weighed crucible with the aid of a clean knife and brush, ignite filter paper separately, add the ash to main portion, ignite for about a quarter of an hour at a red heat in muffle; cool and weigh. The increase in weight of the crucible, minus ash of filter paper = silica (SiO_2), free and combined in 2 grams of sample, which multiplied by 50 = percentage in fuel ash.

The *filtrate* is now transferred to a 400 cc. graduated stoppered cylinder and diluted to 400 cc. and thoroughly mixed: 200 cc. (= 1 gram sample) are poured off into a 300 cc. beaker, the solution boiled, 20 cc. of an E solution of Baric chloride added, stirred, and allowed to stand for at least twelve hours, preferably in a warm place, after which the Baric sulphate ($Ba\,SO_4$) is filtered off and determined as directed on page 7; the $Ba\,SO_4$ found × ·412 × 100 = percentage of sulphuric acid (SO_4) present in ash. The remaining 200 cc. of solution in cylinder are transferred to a 400 cc. beaker, 5 cc. of a saturated solution of ammonium chloride (Am Cl) added, and 20 E ammonic hydrate (Am Ho ·880) run in until the solution shows a strong alkaline reaction; the oxide of iron and alumina are thus precipitated as hydrates and phosphates.

The solution is now gently boiled until the odour of the ammonia has nearly all disappeared; and then filtered through a filter of convenient size, and any particles of precipitate adhering to sides of beaker are detached with the aid of a feather and a jet of water. The precipitate is now washed with boiling water until, when portions of the washings collected in a small test tube, and made acid with HNO_3, show no opalescence with a few drops of nitrate of silver. Reserve the filtrate, which now contains the lime and magnesia as chlorides, detach filter and spread out on a watch-glass, dry in water-oven, scrape precipitate into weighed crucible, ignite filter separately, add ash to main portion and ignite cautiously to a full red heat, cool in desiccator and weigh. Increase in weight *minus* ash $= Fe_2 O_3 + Al_2 O_3 + P_2 O_5$. It now becomes necessary to estimate the oxide of iron, which is best done volumetrically, and from a knowledge of the amount of phosphoric acid ($P_2 O_5$) present, as determinated in another portion of sample (*vide* page 111), the amount of alumina present is obtained. A direct process for the determination of alumina in presence of oxide of iron consists in dissolving out the former in a strong solution of potash, acidulating the solution with HCL and precipitating alumina with ammonic hydrate. The writer has obtained rather better results, however, with the indirect process.

Estimation of Oxide of Iron volumetrically.—Dissolve the precipitate of $Fe_2O_3 + Al_2O_3 + P_2O_5$ off the filter with about 10 cc. of strong hydrochloric acid, collecting the solution in a small porcelain dish; wash the filter till free from iron, salt, &c., with water; evaporate solution over a water bath nearly to dryness to expel excess of free HCL. Transfer to a bulb flask of about 500 cc. capacity, supplied with a rubber cork into which a tube with a Bunsen valve is fitted. Dilute to

200 cc., then add 10 cc., of a 25 per cent. solution of sulphite of soda ($Na_2S_2O_3$) and 1 cc. of 10 E HCL. Attach cork and tube and boil until free from SO_2, which usually takes about a quarter of an hour. Close the valve quickly and put the flask into cold water to cool. The iron is thus reduced from the form of perchloride (Fe_2Cl_6) to that of dichloride ($FeCl_2$), according to the following equation :—

$$Fe_2Cl_6 + OH_2 + Na_2SO_3 = 2\ FeCl_2 + Na_2SO_4 + 2\ HCL.$$

The next step in the process is to ascertain how much of an oxidizing agent is necessary to convert the $FeCl_2$ back again into Fe_2Cl_6. In this case bichromate of potash ($K_2Cr_2O_7$) is the most suitable to use.

When the solution is cold pour it into a beaker of about 500 cc. capacity, and test to ascertain if perfectly reduced by bringing a drop of the solution, on the end of a glass rod, in contact with a drop of a solution of potassic sulphocyanate (KCyS) on a porcelain plate, when if only a mere trace of Fe_2Cl_6 is present, a pink coloration will be produced ; if no coloration, or just the slightest colour is formed, add 10 cc. of hydrochloric acid, sp. gr. 1·16. Fill a burette graduated in 0·1 of 0·cc. with *standard* $\frac{N}{10}$ potassic bichromate ($K_2Cr_2O_7$), prepared by dissolving 4·917 grams of the pure, dry, recrystallized salt in about 500 cc. of pure distilled water, and diluting to 1,000 cc. at 15·5° C.

Run in the $\frac{N}{10}$ $K_2Cr_2O_7$ into the iron solution at the rate of about $\frac{1}{10}$ cc. per second with continual stirring. A drop of the solution is taken out from time to time on the end of a glass rod, and mixed with a drop of dilute solution of (about 1 per cent.) potassic ferricyanide, when a more or less blue coloration is produced as long as there is any ferrous salt present. When no blue or green colour is produced with the ferricyanide on allowing to stand for four minutes the reaction may be considered completed. Note the number of cc. required.

The following is the equation representing the action of the bichromate :—

$$6\,FeCl_2 + 14\,HCl + K_2Cr_2O_7 = 3\,Fe_2Cl_6 + Cr_2Cl_6 + 2\,KCl + 7\,OH_2.$$

Now every cubic centimeter of $\frac{N}{10}$ $K_2Cr_2O_7$ = ·0056 gram of iron or ·008 gram of peroxide of iron (Fe_2O_3). So that the number of cc. of $\frac{N}{10}$ $K_2Cr_2O_7$ required for oxidation multiplied ·008 × 100 = percentage of peroxide of iron in the ash.

The amount of *alumina* present in the mixed oxides can now be obtained by difference, by adding together the Fe_2O_3 present and the P_2O_5 found in another portion of sample, and subtracting from the weight of $Al_2O_3 + Fe_2O_3 + P_2O_5$ found above, and multiplying the remainder by 100, the percentage of alumina is obtained.

Determination of Lime and Magnesia.—To the reserved filtrate containing these bases add 10 cc. of 20 E ammonic hydrate and 10 cc. of a saturated solution of oxalate of ammonium; heat to boiling point, and allow to stand a few hours; filter off oxalate of lime and wash with water till free from chlorides. The filtrate is now put to evaporate over water bath in a platinum dish. The filter and its contents are dried in a water oven, then placed in a weighed crucible and carefully ignited to a bright red heat in a muffle, for about a quarter of an hour. The oxalate of lime (CaC_2O_4) is thus converted into caustic lime (CaO); cool in desiccator and weigh. Increase in weight minus filter ash, multiplied by 100 = percentage of lime present.

When the contents of the platinum dish have evaporated to dryness over the water bath, it is heated carefully by the flame of a Bunsen burner until the whole of the ammoniacal salts are expelled; the dish is then cooled, the residue moistened with about 2 cc. of dilute HCL (1 to 2), about 50 cc. of distilled water added, and the dish heated until all goes into solution. The solution is now transferred to a beaker of about 100 cc. capacity, add 20 cc. of 20 E ammonic hydrate added, together with 15 cc. of a saturated solution of disodic phosphate ($NaHPO_4$) and well stirred and allowed to stand 24 hours; after which the ammonio-phosphate of magnesia is filtered off, and any precipitate adhering to sides of beaker is detached by the aid of a feather and portions of the filtrate. The precipitate is now washed with dilute ammonia solution (1 to 2) till free from chlorides, and the volume of the filtrate measured. The precipitate is soluble to the extent of 1 mgm. in every 50 cc., and this must be added to the total weight. The filter and its contents are spread out on a watch-glass, dried in a water oven, the precipitate brushed into a weighed crucible, the filter paper ignited separately and the ash added to main portion. The crucible is gradually heated in a muffle to a bright red heat for about a quarter of an hour, after which it is cooled in a desiccator and the pyrophosphate of magnesia ($Mg_2P_2O_7$) thus produced weighed. The weight of $Mg_2P_2O_7$ multiplied by ·3604 × 100 = percentage of magnesia (MgO) in ash.

Determination of Phosphoric Acid (P_2O_5).—Weigh out 0·5 gram of the ash into a 50 cc. porcelain dish, pour on 20 cc. of aqua regia (10 EHCL, 10 cc. + 16 E HNO_3 10 cc.) and gently boil with cover on until all that is soluble is dissolved. The solution is now evaporated to dryness, and the residue heated on a sand bath to 130° C. to render silica insoluble; it is now cooled, 10 cc. of strong HCL added, and boiled until all that is soluble is dissolved; add 20 cc. of water, filter off silica, &c., and wash until free from iron salt. To the filtrate add 10 cc. of 16 E nitric acid, and evaporate nearly to dryness on a water bath; dissolve residue up in 6 cc. 16 E nitric acid and 4 cc. of water, dilute to 15 cc. and add 30 cc. of ammonic molybdate reagent (this is prepared by dissolving 55·5 grams of molybdenum trioxide (MoO_3) in a mixture of 94 cc. ammonic hydrate, sp. gr. ·880, and 150 cc. of water, and filtering into 694 cc. of nitric acid, sp. gr. 1·2, diluting to 1,000 cc. with a 12 per cent. solution of HNO_3, allowing to stand for 12 hours at 50° C., filtering and bottling); allow to stand for 3 hours at 40° C. with occasional stirring. Filter, and wash first with a 3 per cent. solution of nitric acid until free from iron salts, and then with water until free from acid (test the solution to ascertain if precipitation was complete by adding 10 cc. more of molybdate reagent to the filtrate and again digesting at 40° C., when if any additional precipitate is formed, the solution is again filtered). Spread the filter on a watch-glass and dry in water oven, brush off the yellow precipitate of phospho-molybdate of ammonium into a weighed crucible, and dry at 100° C. until weight remains constant. The precipitate as dried at 100° C. contains 3·733 per cent. of P_2O_5, so that multiplying the weight found by ·0373 × 2 × 100 the percentage of phosphoric in the ash is obtained.

Determination of Potash and Soda.—Weigh 2 grams of sample into a 100 cc. beaker, add 20 cc. of (10 E) hydrochloric acid, and boil until all that is soluble is dissolved. Evaporate to dryness, and heat residue on sand bath to 130° C. for some time; cool, add to 10 cc. of 10 E HCL, boil, dilute to 80 cc., filter, and wash silica, &c., until free from iron salts, &c. The oxide of iron, alumina, and lime are separated from the solution by the methods described above; the filtrate from the lime determination is evaporated to dryness in a platinum dish, and the residue carefully ignited until no more fumes of ammoniacal salts are evolved. Cool, dissolve residue in 10 cc. of water, add 1·5 grams of oxalic acid, evaporate to dryness as before, and ignite. The alkalies are now left as carbonates, and the magnesic chloride is converted into magnesia (MgO). Treat the residue with 20 cc. of hot water, filter, and wash with water. The filtrate is now transferred to a weighed platinum dish, acidulated with HCL, evaporated to dryness without loss, and the residue carefully heated to dull redness for 10 minutes. Transfer to a desiccator to cool, and weigh the NaCl + KCl. Add 10 cc. of water to residue, and then sufficient platinic chloride ($PtCl_4$) to convert the sodic and potassic chlorides into double chlorides of the alkalies and platinum. Assuming that the whole of the residue is NaCl, a calculation of the quantity required can be made. Thus every 117 parts of NaCl require 336·38 parts of $PtCl_4$, forming $PtCl_4$, 2 NaCl. The mixture is evaporated over a water bath at a temperature of about 90° C., *nearly* to dryness, after which about 15 cc. of alcohol, sp. gr.

·86, are added, the dish covered with a glass plate, and allowed to stand for three hours, with occasional rotatory stirring of the contents. Allow to settle before filtering, decant the clear liquid through a weighed filter, and finally filter off the insoluble double chloride of potassium and platinum with the aid of the filtrate and a feather. Wash with alcohol until free from the $PtCl_4$ 2 $NaCl$; dry at 130° C., cool between two weighed watch-glasses, and weigh. Calculation of the proportion of Na_2O and K_2O present: $PtCl_4$ 2 $KCl \times \cdot 3070 = KCl$, and $KCl \times \cdot 6317 \times 50 =$ percentage of K_2O. Substract the weight of KCl thus calculated from the weight of the $NaCl + KCl$. The remainder $\times \cdot 5302 \times 50 =$ percentage of the Na_2O present.

Determination of Carbonic Acid (CO_2.)—Carbonic acid is not always present in the ash of fuel. When it is present it is combined with potash and soda, or, in some cases where the heat has not been sufficiently intense, it is combined with lime or magnesia. The most accurate method for the determination of carbonic acid consists in decomposing the carbonate present by means of hydrochloric acid, and weighing the liberated CO_2, which may be absorbed by soda, lime, or caustic soda. The following are details of the process, and Fig. 47 is a sketch of the apparatus employed. Weigh out 5 grains of the sample into the flask D, and connect in the manner as shown in sketch. A is a U-tube containing pieces of caustic soda, which serve to absorb the CO_2, contained in the air, that is made to pass through the apparatus by means of the aspirator I. C is a bulb tube containing about 50 cc. of dilute hydrochloric acid, containing 80 per cent. of strong HCL, which is prevented from flowing out by means of a clip at B. E are bulbs containing a little concentrated sulphuric acid, which serves to absorb the great bulk of moisture coming from D. F is a U-tube containing in the first limb pieces of pumice-stone which have been soaked in a concentrated solution of sulphate of copper, dried and ignited; this serves to absorb any HCL carried over with the gas; the second limb contains pieces of anhydrous calcium chloride. G is a U-tube containing in the first three-quarters small pieces of caustic potash, and the remaining quarter pieces of anhydrous calcium chloride. This latter tube absorbs the CO_2 given off from the sample, which is to be weighed before and after the experiment. H is a tube containing pieces of calcium chloride, which serve to absorb any moisture from the air that may tend to come into G from the aspirator I, which is to be nearly filled with water. When all is fixed and the tube G accurately weighed, open the clip B and allow the acid to run on to the ash in D; after which open the tap K, and allow a gentle stream of water to flow out; a current of air thus passes through the apparatus and sweeps the CO_2 out of D, and which is absorbed in the U-tube G. Continue the aspiration in the cold until no more effervescence is observed, and then apply a gentle heat to the flask D; continue heating to incipient ebullition, until it is assured that all CO_2 is eliminated, when the tube G is detached and weighed. The increase in weight $\times \frac{100}{6}$ is the percentage of combined CO_2 present.

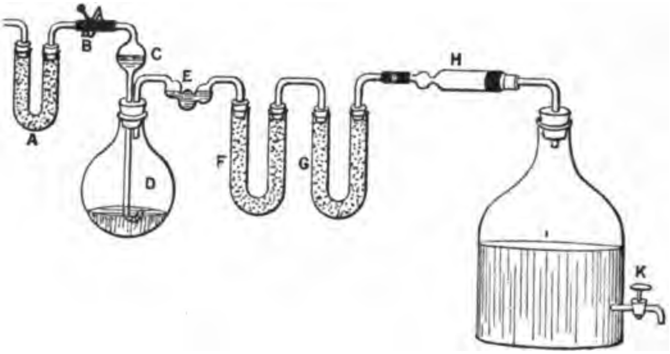


FIG. 47.

Tables of Practical Results and Analyses.

COMPARATIVE CONSUMPTION OF COAL BY COMPOUND AND ORDINARY LOCOMOTIVES FOR THREE MONTHS ENDING 21ST MAY, 1886, ON THE GREAT EASTERN RAILWAY.

Number and Class of Engines.	1886. Four weeks ending.	Distance run.		Coal consumed.		
		Train Miles.	Engine Miles.	Total Cwts.	lbs. per Train Mile.	lbs. per Engine Mile.
Eleven compounds .	26 Mar.	36,503½	37,794¼	9,849	30·2	29·1
Eleven ,, .	23 Apl.	34,848¼	35,857¾	8,979	28·8	28·0
Eleven ,, .	21 May	38,117¾	39,102¾	9,939	29·2	28·4
Totals and averages .	. .	109,469½	112,754¾	28,767	29·4	28·5
Six ordinary . .	26 Mar.	18,610	19,355½	5,724	34·4	33·0
Seven ,, . .	23 Apl.	23,761	24,362¼	7,162	33·7	32·9
Seven ,, . .	21 May	22,300	22,938½	6,661	33·4	32·5
Totals and averages .	. .	64,671	66,656¼	19,547	33·8	32·8
Mean saving by compounds	.	.	.	. .	4·4	4·3

COMPARATIVE CONSUMPTION OF COAL AND EVAPORATION OF WATER BY COMPOUND AND ORDINARY LOCOMOTIVES WORKING PASSENGER TRAINS FROM LONDON TO NORWICH ON THE GREAT EASTERN RAILWAY.

October, 1886.		Compound No. 704.	Ordinary No. 565.
Coal consumption, total .	lbs.	2780	3444
,, ,, per mile .	lbs.	24·3	30·2
Water evaporation, total .	gallons	2196	2853
,, ,, per lb. of coal	lbs.	7·9	8·2
Feed water, average per 5 minutes	gallons	112·5	126·4
Feed water temperature . .	Fahr.	64°	65°
Average steam pressure per square inch . . .	lbs.	138	122
Load, London to Ipswich .	vehicles	14	15
,, Ipswich to Norwich .	vehicles	6	7

The compound steamed freely, weather very favourable.
The ordinary engine steamed moderately, weather rather unfavourable.

AVERAGE COMPOSITION OF COALS FROM DIFFERENT LOCALITIES.

(*Phillips' Admiralty Coal Investigation.*)

Locality (Average Number of Samples from each).	Sp. Gr.	Carbon.	Hydrog.	Nitrog.	Sulph.	Oxyg.	Ash.	Coke.
36 from Wales . .	1·315	83·78	4·79	0·98	1·43	4·15	4·91	72·60
18 from Newcastle . .	1·256	82·12	5·31	1·35	1·24	5·69	3·77	60·67
28 from Lancashire . .	1·273	77·90	5·32	1·30	1·44	9·53	4·88	60·22
8 from Scotland . .	1·259	78·53	5·61	1·00	1·11	9·69	4·03	54·22
7 from Derbyshire . .	1·292	79·68	4·94	1·41	1·01	10·28	2·65	59·32

COMPOSITION OF VARIOUS ANTHRACITES.

Locality.	Sp. Gr.	Carbon.	Hydrogen.	Oxygen, Nitrogen, and Sulphur.	Ash.	Observers.
Pennsylvania . .	1·462	89·21	2·43	3·69	4·67	Regnault.
Swansea . . .	1·348	91·29	2·33	4·80	1·58	
Mayenne . . .	1·343	90·20	4·18	3·37	2·25	
Roldue(Aix-la-Chap.)	1·367	90·72	3·92	4·42	0·94	
Swansea . . .	1·270	90·58	3·60	4·10	1·72	Jacquelin.
Sablé . . .	1·750	87·22	2·49	3·39	6·90	
Vizille . . .	1·730	94·09	1·85	2·85	1·90	
Isere . . .	1·650	94·00	1·49	3·58	4·00	

ANALYSES (BY THE AUTHOR) OF THREE SAMPLES OF ANTHRACITIC STEAM COAL MINED AT TONKIN.

	Sample from Hatou.	Sample from Nagotna.	Sample from Kamfa.
Carbon . . .	88·075	90·575	90·605
Hydrogen . .	4·250	3·459	3·256
Oxygen . . .	3·558	2·367	2·585
Nitrogen . .	1·126	1·010	0·706
Sulphur . .	0·487	2·465	0·350
Ash . . .	2·504	2·134	2·498
	100·000	100·000	100·000

ANALYSES (BY THE AUTHOR) OF STEAM COAL OF THE MONMOUTHSHIRE COALFIELD (EBBW VALE STEEL, IRON, AND COAL CO., LIMITED).

	Three-quarter Vein.	Elled Seam.	Old Coal.	Big Vein.
Carbon	89·055	88·057	87·350	86·956
Hydrogen	4·750	4·652	4·625	4·600
Oxygen	2·014	3·407	3.200	2·874
Nitrogen	1·556	1·055	1·215	1·450
Sulphur	0·875	0·755	0·795	0·815
Ash	1·750	2·074	2·815	3·305
	100·000	100·000	100·000	100·000
Moisture	0·625	0·558	0·560	0·645
Coke	80·050	79·405	81·032	77·050
Volatile matter (gas, tar, &c.)	19·950	20·595	18·968	22·950
Fixed carbon	78·300	77·331	78·217	73·745
Carbon in volatile matter	10·755	10·726	9·133	13·211
Available hydrogen	4·498	4·226	4·225	4·242
Specific gravity	1·295	1·310	1·308	1·315
Volume occupied by 1 ton of coal calculated from sp. gr. (cubic feet)	28·109	27·335	27·400	27·254
THERMAL VALUES:—				
Theoretical calorific power, viz. lbs. of water raised 1° C. by 1 lb. of coal (French calories)	8,745	8,575	8,514	8,487
British thermal units, viz. lbs. of water raised 1° F. by 1 lb. coal	15,741	15,435	15,325	15,276
Evaporative power, viz. lbs. of water evaporated from 212° F. by 1 lb. of coal	16·28	15·98	15·85	15·80
Actual evaporation in active boiler practice—lbs. of water converted into steam	11·00	10·55	10·55	10·31

TABLE SHOWING THE PROGRESSIVE DIMINUTION OF HYDROGEN AND OXYGEN FROM WOOD TO ANTHRACITE.

(*Prof. Johnson.*)

	Carbon.	Hydrogen.	Oxygen.	Disposable Hydrogen.
Wood (average)	100	12·18	83·07	1·80
Peat "	100	9·85	55·67	2·89
Lignite (mean of 15 varieties)	100	8·37	42·42	3·07
Coal, South Staffordshire	100	6·12	21·23	3·47
Steam coal, Newcastle	100	5·91	18·32	3·62
Portrefelin coal, S. Wales	100	4·75	5·28	4·09
Pennsylvanian anthracite	100	2·84	1·74	2·63

ANALYSIS OF PATENT FUELS.

(*Admiralty Investigation.*)

Kind of Fuel.	Sp. Gr.	Carbon.	Hydrogen.	Nitrogen.	Sulphur.	Oxygen.	Ash.	Coke.
Warlich's patent fuel .	1·15	90·02	5·56	trace	1·62	—	2·91	85·1
Livingstone's steam fuel .	1·184	86·07	4·13	1·80	1·45	2·03	4·52	—
Livon's patent fuel . .	1·13	86·36	4·56	1·06	1·29	2·07	4·66	—
Wylam's ,, . . .	1·10	79·91	5·69	1·68	1·25	6·63	4·84	65·8
Bell's ,, . . .	1·14	87·88	5·22	0·81	0·71	0·42	4·96	71·7
Holland & Green's patent fuel	1·302	70·14	4·65	1·15	—	—	13·73	—

AVERAGE VALUE OF COALS FROM DIFFERENT LOCALITIES.

Locality.	Lbs. of water evaporated from 100° C. by 1 lb. of coal.	Number of lbs. evaporated per hour.	Weight in lbs. of 1 cubic foot of coal as used for fuel.	Space occupied by 1 ton in cubic feet.	Results obtained in experiments on cohesive power of coals (percentage of large coals).	Per cent. of sulphur in coals.
Average of 37 samples from Wales . .	9·05	448·2	53·1	42·71	60·9	1·42
Average of 17 samples from Newcastle .	8·37	411·1	49·8	45·30	67·5	0·94
Average of 28 samples from Lancashire .	7·94	447·6	49·7	45·15	73·5	1·42
Average of 8 samples from Scotland .	7·70	431·4	50·0	49·99	73·4	1·45
Average of 8 samples from Derbyshire .	7·58	432·7	47·2	47·45	80·9	1·01

Comparative Values of Welsh Steam Coals.

(*Portsmouth Dockyard Experiments.*)

	Lbs. of water evaporated by 1 lb. of coal.	Percentage of clinker and ash.
Nixon's Navigation .	10·05	5·37
Wayne's Merthyr . .	10·05	5·37
Thomas „ . .	9·79	5·47
Nauhudyn „ . .	9·62	5·48
Ynsfaio	9·52	6·76
Merthyr Dare . . .	9·45	5·48
Resolven Merthyr . .	9·41	6·04
Insoles	9·37	6·52
Averages . .	9·65	5·81

Analyses of the Ash of Seven Varieties of Bituminous Coals.

(*Phillips' Admiralty Coal Investigation.*)

	Name of Coal.	Silica.	Alumina and Oxide of Iron.	Lime.	Magnesia.	Sulphuric Acid.	Phosphoric Acid.	Total Percentage.	Percentage of Ash in Coal.	Percentage of Coke in Coal.
Welsh.	Pontypool .	40·00	44·78	12·00	trace	2·22	0·75	99·75	5·52	64 8
	Bedwas .	26·87	56·95	5·10	1·19	7·23	0·74	98·08	6·94	71·7
	Porthmawr.	34·21	52·00	6·199	0·659	4·13	6·633	97·821	2·91	63·1
	Ebbw Vale.	53·00	35·01	3·94	2·20	4·89	0·88	99·92	14·72	77·5
	Coleshill .	59·27	29·09	6·02	1·35	3·84	0·40	99·97	10·70	—
Scotch.	Splint .	37·60	52·00	3·73	1·10	4·14	0·88	99·45	1·50	52·03
	Wallsend Elgin	61·66	24·42	2·62	1·73	8·38	1·18	99·99	4·00	58·45

ANALYSES OF VARIOUS COKE ASHES.

Locality.	Sulphate of Lime.	Silica.	Alumina.	Carbonate of Lime.	Carbonate of Magnesia.	Oxide of Iron.	Oxide of Manganese.
Iron Bridge . .	12·55	42·10	34·40	4·80	0·40	5·28	trace
Dudley . . .	8·64	35·40	30·40	6·48	—	18·68	,,
Merthyr Tydvil . .	4·54	41·60	35·44	6·46	1·08	10·80	,,

THE SPECIFIC GRAVITIES AND AMOUNTS OF ASH IN VARIOUS COALS.

Description and Locality of Coal Beds.	Specific Gravity.	Percentage of Ash.	Authority.
Liverpool coal . . .	1·260	4·62	Johnson.
Alfreton furnace coal . .	1·235	2·04	D. Musket.
Wigan cannel coal . .	1·272	3·10	Kerwan.
Boghead brown cannel .	1·160	21·2	Dr. Penny.
Stevenston Ayrshire . .	1·385	19·3	
Ayrshire anthracite . .	1·453	3·6	
Aberaman Merthyr . .	1·305	1·45	J. A. Phillips.
Pontypool	1·320	5·52	
Abercarn	1·334	2·04	
Wallsend Elgin . . .	1·200	10·70	
Stavely	1·270	2·4	
Neath Abbey . . .	1·310	3·55	
Ebbw Vale	1·274	3·70	H. J. Phillips.
Blaina	1·320	6·80	
Wharncliffe Woodmoor .	1·294	7·1	
Shelton	1·285	4·2	
Elsegar	1·300	3·4	
Nixon's Merthyr . . .	1·305	1·25	

RESULTS OF EVAPORATIVE DUTIES OF NEWCASTLE AND WELSH COALS IN THE COAL-TESTING MARINE BOILER AT KEYHAM STEAM FACTORY.

Coal.	Area of Fire-grate.	Coal consumed per hour.	Coal per square foot of grate, per hour.	Water consumed from 100° per hour.	Water per sq. foot of grate per hour.	Water evaporated from 100° C. per lb. of coal.
	sq. ft.	cwts.	lbs.	cub. ft.	cub. ft.	lbs.
FIRST SERIES. (With common doors.)						
Welsh, Wayne's Merthyr, Resolven, Merthyr Dare, Gellia, Cadoxton	14·0	1·93	15·44	32·4	2·31	10·42
Hartley Main Newcastle	14·0	2·32	18·56	34·5	2·46	9·22
½ Welsh ½ Hartley	14·0	1·92	15·40	30·4	2·17	9·81
2 " 1 "	14·0	1·76	14·08	28·7	2·05	10·12
1 " 2 "	14·0	1·96	15·70	30·7	2·20	9·72
(With perforated doors.)						
Hartley Main	14·0	2·06	16·50	30·2	2·16	9·10
SECOND SERIES. (With common doors.)						
Welsh—Powell's Duffryn, Nixon's Navigation, Davis's Merthyr	14·0	2·09	16·68	37·1	2·65	11·05
Newcastle — Davidson's Hartley, Hasting's Hartley	14·0	2·29	18·29	34·5	2·46	9·39
½ Welsh ½ Hartleys	14·0	2·03	16·24	34·4	2·46	10·56
2 " 1 "	14·0	2·43	16·36	35·0	2·50	10·61
Welsh	14·0	2·19	17·48	39·3	2·80	11·16
THIRD SERIES. (With perforated doors.)						
Welsh coal	14·0	1·87	14·95	32·7	2·34	10·86
Hartleys	14·0	2·13	17·04	32·8	2·34	9·61
½ Welsh ½ Hartleys	14·0	2·18	17·44	37·1	2·65	10·54
2 " 1 "	14·0	2·08	16·64	35·7	2·55	10·64
1 " 2 "	14·0	2·18	17·42	36·5	2·61	10·39
Davidson's Hartley	14·0	2·86	22·88	42·9	3·06	9·31
½ Hartley ½ Welsh	14·0	2·30	18·40	31·0	2·22	10·80
FOURTH SERIES. (With smaller grate area. With common doors.)						
Welsh coal	10·5	2·11	22·46	38·3	3·65	11·31
½ Welsh small ½ Davidson's Hartley	10·5	2·02	21·60	36·0	3·43	11·06
½ Welsh beans ½ Hasting's Hartley	10·5	2·14	22·85	36·7	3·50	10·65
FIFTH SERIES. (With perforated doors.)						
Hartleys	10·5	2·29	24·40	42·0	4·00	11·42
½ Welsh ½ Davidson's Hartley	10·5	2·10	22·34	39·3	3·74	11·65

DISPOSAL OF HEAT OF COMBUSTION OF COAL BURNT UNDER STEAM BOILERS, AND COMPOSITION OF THE PRODUCTS OF COMBUSTION.

The following are results obtained by MM. Scheurer-Kestner and Meunier in 1868 in a French boiler at Thann. (*Vide Bulletin de la Société Industrielle de Mulhouse.*)

	sq. ft.
Heating surface of heaters	301·3
„ „ boiler	129·1
Total of boiler	430·4
Heating surface of feed heaters	764·0
	1,194·4
Direct heating surface exposed to fire	32·3
Area of fire-grate	19·3
Area of air-spaces through grate	5·5
Ratio of grate area to total surface of boiler	1 to 22·3
Ratio of grate area to total surface of boiler and fuel heaters	1 to 63

The average composition of the Ronchamp coal used in the experiments devoted to the analysis of the chimney gases was as follows :—

Carbon	70
Hydrogen	4
Oxygen	4
Nitrogen	1
Ash	21
	100

ANALYSIS OF THE PRODUCT OF COMBUSTION OF RONCHAMP COAL UNDER A FRENCH BOILER AT THANN.

No. of Experiments.	Coal per sq. ft. grate per hour.	Weight of each Charge.	Intervals of Charges.	Composition of the Gases.						Total Air per lb. of Coal.
				Carbonic Acid.	Carbonic Oxide.	Vapour.	Hydrogen.	Nitrogen.	Free Air.	
	lbs.	lbs.	mins.	p. c.	p. c.	p. c.	p. c.	p. c.	p. c.	cu. ft.
12	8·2	15·4	5	14·9	·84	1·15	1·35	75·1	6·7	110·0
11	9·6	30·8	8	14·2	·97	1·11	1·11	72·5	10·5	116·2
9	9·6	15·4	4	14·6	·86	·56	·56	70·1	13·3	134·7
14	8·2	30·8	10	13·4	·24	1·41	1·41	63·7	20·9	144·5
13	8·2	15·4	5	13·3	—	·91	·91	67·7	17·6	147·5
8	4·7	15·4	8	12·9	—	·96	·96	59·7	26·2	156·6
10	19·0	15·4	2	10·9	—	·19	·19	45·9	42·8	198·3
7	3·4	13·2	10	8·2	—	0·4	·52	37·4	53·8	260·5

CARBON AND HYDROGEN IN ESCAPED COMBUSTIBLE GASES.

No. of Experiments.	Per cent. of Total Carbon.			Per cent. of Total Hydrogen.	Temperature of Gases leaving Feed heaters.
	In Oxide.	In Hydro-carbons.	Total.		
12	4·1	11·4	15·6	19·5	119° C.
11	5·0	10·2	15·3	16·7	128
9	5·2	5·9	11·2	9·9	126
14	1·5	4·1	5·7	26·9	—
13	—	—	6·1	17·5	135
8	—	—	3·9	19·7	93
10	—	—	3·4	4·7	156
7	—	—	·9	17·7	94

It appears from these results that the most effective combination is arrived at when about one-third of the gaseous products consists of free air.

Influence of Excess of Air.—The following table shows the influence of excess of air in using Friedrichsthal and Altenwald coal:—

Friedrichsthal Coal.		Altenwald Coal.	
Free Air. Per cent.	Lbs. Water Evap. per lb. of Coal.	Free Air. Per cent.	Lbs. Water Evap. per lb. of Coal.
40	6·80	35	7·06
36	6·46	33	7·28
30	6·38	32	7·02
27	6·19	30	6·79
27	6·23	28	6·85
24	5·68	25	6·71
23	5·80	23	6·66

Distribution of the Heat of Combustion.—For the comparison of the absolute heat of combustion of coals, the boiler, fire-grate, &c., in this case had the following dimensions:—

	sq. ft.
Area of fire-grate 4·6 feet long by 4·47 feet wide . .	20·6
Ratio of grate to heating surface of boiler . . .	1 to 21
" to total service of boiler and feed heaters	1 to 58
	cu. ft.
Total capacity of boiler	423·6
" " feed heaters	317·7
Water room in boiler	335·3
Steam room in boiler	88·3
	sq. ft.
Heated surface of brickwork for conduction and radiation of heat . . . . above	1,290

The following are the results obtained with various coals in the above boiler:—

Coal.	Observed Total Heat of Combustion. Units.	Air at 17° C. per lb. of Coal. Cu. ft.	Free Air. Per cent.	Temperatures.			Ash.	Pressure of Steams. Atmosphere.	Water per lb. of Coal from and at 100° C.
				Air. °C.	Feed Heater. °C.	Smoke. °C.			lbs.
Ronchamp, No. 3 .	7,825	152·3	24·7	17	65	132	17·3	4·46	8·77
,, No. 4 .	7,775	160·7	29·1	21	71	138	15·8	4·84	9·49
Sarrebrück (mean of 7 coals)	7,500	159·0	31·0	20	72	130	14·0	4·72	8·17
Blanzy, Montceau .	7,067	135·4	23·9	18	65	160	12·0	4·60	7·89
,, anthracite .	7,125	152·3	30·5	16	64	175	24·4	4·58	8·18
Creusot, anthracite .	8,949	269·0	47·6	—	72	144	9·1	4·60	10·53
Creusot, ⅔ ; Ronchamp, ⅓ . . .	8,565	230·1	36·2	11	67	132	13·4	4·71	10·54
Creusot, ⅔; Ronchamp, ⅓. . . .	8,630	214·9	34·2	8	62	145	15·9	4·71	9·83
Wood, charcoal . .	8,080	250·4	42·5	—	—	155	0·5	—	9·2

The absolute heat of combustion of the fuels was estimated from the above to have been distributed about as follows:—

	per cent.
Heat in the steam (about 60 lbs. pressure)	61·0
Heat ungenerated in the combustible gases	5·5
Heat lost in the clinker and ash	1·5
Heat carried off in the gaseous products of combustion .	5·5
Heat ungenerated in the smoke carbon	·5
Heat absorbed in the evaporation of the hygrometric water, and water newly formed	2·5
Heat lost in the brickwork	23·5
Total	100·0

RESULTS OBTAINED WITH LIQUID FUEL BY MR. JAMES HOLDEN, M.I.C.E., ON THE GREAT EASTERN RAILWAY.

EXPERIMENT ON A SMALL CORNISH BOILER.

Coal only used.

1887. Consumption during one week from August 15th to

20th (inclusive), 74¼ hours' work, including lighting up = 80½ cwt. = 121·3 lbs. per hour.

Cost for 100 hours = 12,130 lbs. of coal at 11s. per ton = £2 19s. 7½d.

Coal, Coke, and Tar used. Holden's System.

1888. Consumption during one week from June 25th to 30th (inclusive), 87¾ hours working; including lighting up

= coal 15 cwt. = 19·2 lbs. per hour
= coke 11½ cwt. = 14·7 ,, ,,
Gas tar 280 gallons = 35·1 ,, ,,
Total 69·0 ,, ,,

Cost for 100 hours

= 1,920 lbs. of coal at 11s. per ton = 0 9 5½
= 1,470 lbs. of coke at 9s. 6d. ,, = 0 6 1½
= 3,510 lbs. of tar at 12s. 6d. ,, = 0 19 7¼
Total £1 15 2¼

Comparative Cost of Liquid Fuel and Coal on Locomotives of the Great Eastern Railway.

No. of Engine.	Total Miles run.	Total lbs. used.			Lbs. per Mile.			Total lbs. of Coal, Liquid Fuel, and Chalk per mile.	Proportion of Liquid Fuel and Chalk to Coal used.	Total cost of Fuel.	Cost per mile in Pence.
		Coal.	Liquid Fuel.	Chalk.	Coal.	Liquid Fuel.	Chalk.				
									per cent.	£ s. d.	
193	951¾	13,511	10,505	784	14·2	11·0	8	26·0	83	9 1 5	2·28
194	951¾	27,738	—	—	29·1	—	—	29·1	—	9 4 10½	2·33
Difference in favour of No. 193 engine . .										0 3 5½	·05

N.B. Cost of coal computed at 14/11 per ton (Radford coal used).
,, liquid fuel computed at 1⅛d. per gallon of 11 lbs.
,, chalk computed at 5/6 per ton.

COMPARATIVE EVAPORATION OF WATER BY DIFFERENT LIQUID FUELS INJECTED AND SPRAYED WITH HOLDEN'S PATENT INJECTOR.

			Evaporative power of Liquid Fuel alone.
ON A SMALL VERTICAL BOILER ABOUT 6-H.P. WORKING AT 50 LBS. PER SQUARE INCH PRESSURE.			
With Yorks coal		4·7 lbs. water per lb. of fuel	
,, ,,	and coal tar	5·7 ,, ,, ,,	6 lbs.
,, ,,	and green oil	6·6 ,, ,, ,,	7·3 ,,
,, ,,	and astatki	9·1 ,, ,, ,,	10·2 ,,
ON A SMALL CORNISH BOILER ABOUT 30-H.P. WORKING AT A PRESSURE OF 35 LBS. PER SQUARE INCH.			
With Yorks coal		8·4 lbs. water per lb. of fuel	
,, ,,	and coal tar	9·9 ,, ,, ,,	11·3 lbs.
,, ,,	and green oil	10·3 ,, ,, ,,	12·3 ,,
,, ,,	and astatki	12·3 ,, ,, ,,	14·5 ,,
ON A LOCO. BOILER WORKING AS STATIONARY AT 80 LBS. PER SQUARE INCH PRESSURE.			
With Yorks coal		9·1 lbs. water per lb. of fuel	
,, ,,	and green oil	12·9 ,, ,, ,,	15·1 lbs.

In all cases the figures give evaporation from feed water at atmospheric temperature.

Shale oil about equals green oil for evaporative value.

COMPARATIVE CONSUMPTION OF COAL AND OIL IN ORDINARY AND COMPOUND PASSENGER LOCOMOTIVES ON THE BUENOS AYRES AND ROSARIO RAILWAY.

Four weeks from 27 May to 25 June, 1887.	Distance run.	Coal, lbs.		Oil, lbs.	
		Total.	Per Mile.	Total.	Per 100 Miles.
Ordinary engine, No. 10.	3,937	111,001	28·19	595·24	15·12
Compound ,, No. 34.	3,638	81,350	22·36	599·65	16·48

Thus the compound burnt 20½ per cent. less coal than the ordinary engine, but used 9 per cent. more oil; the actual money saving was 3 dollars 20 cents per hundred miles. The working pressure in the ordinary engine was 150 lbs. per square inch, and in the compounds 160 lbs.

The following are very instructive results obtained by Mr. Urquhart on the Grazi and Tsaritsin Railway locomotives, showing the efficiency of petroleum refuse:—

COMPARATIVE TRIALS WITH PETROLEUM, ANTHRACITE, BITUMINOUS COAL, AND WOOD, BETWEEN ARCHEDA AND TSARITSIN ON GRAZI AND TSARITSIN RAILWAY IN WINTER.

Train alone.		Distance run.	Car. Miles.	Fuel.	Consumption, including Lighting-up.		Cost of Fuel per Train Mile.	Atmospheric Temperature and Weather.
No. of Loaded Cars.	Gross Load.				Total.	Per Train Mile.		
	tons.	miles.			lbs.	lbs.	pence.	
25	400	388	9,700	Anthracite .	31,779	81·90	11·957	—14° to —13° C. Strong side wind.
25	400	388	9,700	Bituminous coal .	37,557·5	96·53	14·093	
25	400	194	4,850	Petroleum refuse	9,462	48·77	5·487	
25	400	194	4,850	Anthracite .	12,639·5	65·15	9·512	—6° to —11° C. Light side wind.
25	400	194	4,850	Wood in billets	Cubic ft. 1,071·8	Cu. ft. 5·52	8·5	
25	400	194	4,850	Petroleum refuse	lbs. 7,223	lbs. 37·23	4·188	

Prices of Fuel:—Petroleum refuse, 21s. per ton.
Anthracite and bituminous coal, 27s. 3d. per ton.
Wood, 1·47d. per cubic foot.

Dimensions of Locomotives:—Cylinders, 18⅛" dia. and 24" stroke. Wheels, 4 ft. 3 in. dia. Total heating surface, 1,248 sq. ft. Total adhesion weight, 36 tons. Boiler pressure, 8 to 9 atmos.

COMPARATIVE MONTHLY AVERAGES DURING 1883 WITH COAL AND PETROLEUM REFUSE IN LOCOMOTIVES, WORKING MAIN LINE TRAINS ON GRAZI AND TSARITSIN RAILWAY.

CONSUMPTION OF FUEL PER TRAIN MILE.

Locomotives.	Trains.	Fuel.	Monthly Averages of Consumption per Train Mile in lbs.												Mean.
			Jan.	Feb.	Mar.	April.	May.	June.	July.	Aug.	Sept.	Oct.	Nov.	Dec.	
Eight wheels coupled	Goods	Coal .	98·06	108·96	100·79	76·27	76·27	79·00	74·91	73·55	79·00	85·81	98·06	95·34	87·17
Six wheels coupled	Goods	Coal . .	73·55	77·63	70·82	64·01	55·84	61·29	54·48	55·84	65·38	80·36	92·62	85·81	69·80
		Petroleum refuse	53·12	54·48	46·31	42·22	34·05	35·41	31·33	36·10	40·86	39·50	50·39	54·48	43·19
Four wheels coupled	Mixed .	Coal . .	51·76	76·27	43·58	34·05	36·77	35·41	42·22	49·03	51·76	40·86	49·93	58·57	47·44
Four wheels coupled	Passenger	Coal . .	40·86	49·03	46·31	36·77	34·05	32·69	31·33	32·69	36·77	39·50	42·22	50·39	39·38
		Petroleum refuse	—	—	—	—	—	—	—	—	20·43	31·33	32·69	34·05	29·62

COST OF FUEL PER TRAIN MILE.

Locomotives.	Trains.	Fuel.	Monthly Averages of Cost per Train Mile in Pence.												Mean.
			Jan.	Feb.	Mar.	April.	May.	June.	July.	Aug.	Sept.	Oct.	Nov.	Dec.	
Eight wheels coupled	Goods	Coal . .	13·495	15·635	14·346	11·226	11·319	11·704	11·186	10·925	11·552	12·471	14·600	13·930	12·699
Six wheels coupled	Goods	Coal . .	10·520	11·099	9·897	9·405	8·387	9·317	8·217	8·279	9·694	11·776	13·647	12·344	10·212
		Petroleum refuse	7·294	7·602	6·158	4·973	4·040	4·163	3·617	4·170	4·948	4·771	6·817	7·356	5·495
Four wheels coupled	Mixed .	Coal . .	7·35[illegible]	10·973	6·672	4·934	5·466	4·988	6·267	7·269	11·215	5·908	7·102	8·652	6·932
Four wheels coupled	Passenger	Coal . .	6·070	7·023	6·602	5·144	4·829	4·670	4·445	4·819	5·165	5·633	6·111	7·580	5·672
		Petroleum refuse	—	—	—	—	—	—	—	—	2·411	5·611	4·366	4·634	3·808

Of the Coal consumed 49 per cent. was anthracite and 51 per cent. was bituminous coal.

Mean Price of Fuel:—Petroleum refuse, 23s. 4d. per ton; Coal, 27s. per ton.

The following results were obtained on the above line in summer time:—

Train alone.		Train Miles.	Fuel.	Consumption, including Lighting-up.		Cost of Fuel per Train Mile.
No. of Loaded Cars.	Gross Load.			Total.	Per Train Mile.	
No.	Tons.			lbs.	lbs.	pence.
30	480	194	Bituminous coal	14,084·07	72·598	10·599
30	480	194	Petroleum refuse	6,175·325	31·831	3·581
30	480	194	Anthracite .	12,784·002	65·897	9·621
30	480	194	Petroleum refuse	6,103·097	31·459	3·539

Theoretical Evaporative Value of Petroleum Fuel and Coal.

Fuel.	Specific Gravity at 32° F.	Chemical Composition.				Heating Power. British Thermal Units.	Theoretical Evaporation. Lbs. of Water per lbs. of Fuel.	
		Carbon, per Cent.	Hydrogen, per Cent.	Oxygen, per Cent.	Sulphur, per Cent.		From and at 212° F.	At 8½ Atmospheric effective Pressure.
							lbs.	lbs.
Pennsylvanian heavy crude oil	·886	84·9	13·7	1·4	—	20,736	21·48	17·8
Caucasian light crude oil .	·884	86·3	13·6	0·1	—	22,027	22·79	18·9
Caucasian heavy crude oil .	·938	86·6	12·3	1·1	—	20,138	20·85	17·3
Petroleum refuse .	·928	87·1	11·7	1·2	—	19,832	20·53	17·1
Good English coal, mean of 98 samples . .	1·380	80·0	5·0	8·0	1·25	14,112	14·61	12·16

In comparing petroleum refuse (Russian) and anthracite, Mr. Urquhart says that "the former has a theoretical evaporative power of 16·2 lbs. of water per lb. of fuel, and the latter of 12·2 lbs., at an effective pressure of 8 atmospheres, or 120 lbs. per square inch; hence petroleum has, weight for weight, 33 per cent. higher evaporative value than anthracite. Now in locomotive practice, a mean evaporation of from 7 to 7½ lbs. of water per lb. of anthracite, is about what is generally obtained; thus giving about 60 per cent. of efficiency, while 40 per cent. of the heating power is unavoidably lost. But with petroleum an evaporation of 12·25 lbs. is practically obtained, giving $\frac{12{\cdot}25}{16{\cdot}2}$ = 75 per cent. efficiency. Thus, in the first place, petroleum is theoretically 33 per cent. superior to anthracite in evaporative power, and, secondly, its useful effect is 15 per cent. greater, being 75 per cent. instead of 6 per cent.; while, thirdly, weight for weight, the practical evaporative value of petroleum must be reckoned as at least from $\frac{12{\cdot}25 - 7{\cdot}50}{7{\cdot}50} =$ 63 per cent. to $\frac{12{\cdot}25 - 7{\cdot}00}{7{\cdot}00} =$ 75 per cent. higher than that of anthracite.

Analyses of Various Gaseous Fuels.

ANALYSIS OF SIEMENS PRODUCER GAS.

(*Trans. Amer. Inst. Min. Eng.*)

	1	2	3	4	5
	per cent.	per cent.	per cent.	per cent.	per cent.
Carbonic acid . . .	3·9	8·6	9·3	1·5	6·1
Carbonic oxide . .	27·3	20·0	16·5	23·6	22·3
Hydrogen	—	8·7	8·6	6·0	28·7
Marsh gas	1·4	1·2	2·7	3·0	1·0
Nitrogen	67·4	61·4	62·9	65·9	41·9
Heat units	93966	97184	99074	114939	164164

ANALYSIS OF AMERICAN NATURAL GAS.

(*Ford, Jr. I. and S. Inst.*)

	1	2	3	4	5
	per cent.	per cent.	per cent.	per cent.	per cent.
Carbonic acid . . .	0·8	0·6	nil.	0·4	nil.
Carbonic oxide . .	1·0	0·8	0·58	0·4	1·00
Oxygen	1·1	0·8	0·78	0·8	2·10
Olefiant gas . . .	0·7	0·8	0·98	0·6	0·80
Ethylic hydride . .	3·0	5·5	7·92	12·3	5·20
Marsh gas	72·18	65·25	60·70	49·58	57·85
Hydrogen	20·02	26·16	29·03	35·92	9·64
Nitrogen	nil.	nil.	nil.	nil.	23·41
Heat units	728746	698852	627170	745813	592380

ANALYSIS OF THE GASES AT DIFFERENT DEPTHS OF THE ALFRETON BLAST FURNACE.

	Distance below the Furnace Mouth.				
	8 feet	14 feet	20 feet	24 feet	34 feet
Nitrogen	54·77	50·95	60·46	56·75	58·05
Carbonic acid . . .	9·42	9·10	10·83	10·08	—
Carbonic oxide . .	20·24	19·32	19·48	25·19	37·43
Marsh gas	8·23	6·64	4·40	2·33	—
Hydrogen	6·49	12·42	4·83	5·65	3·18
Olefiant gas . . .	0·85	1·57	—	—	—
Cyanogen	—	—	—	trace	1·34
	100·00	100·00	100·00	100·00	100·00

ANALYSIS OF WATER-GAS.

	(1) "Strong" Gas (Dr. Moore).	(2) Water-Gas Coke used (Langlois).	(3) Water-Gas Coke used (Frankland).	(4) Gas made by Beilby's Process.
Carbonic acid . . .	2·05	12·000	13·80	21·32
Carbonic oxide . .	35·88	31·860	29·30	10·72
Marsh gas	4·11	1·62	56·9	nil.
Hydrogen	52·76	54·52		37·19
Oxygen	0·77	—	—	—
Nitrogen	4·43	—	—	30·77
	100·00	100·00	100·00	100·00

GASES OCCLUDED IN COAL.

(*Thomas.*)

Sample of Coal.	Gas evolved by 100 grams at 100° C. in a vacuum cc.	CO_2.	CH_4.	C_2H_6.	$(C_2H_5)_2$.	N.
Wigan cannel, 5/3 seam, 350 yards deep	421·3	6·44	80·69	4·75	—	8·12
Do., 3/2 seam, 600 yards deep	350·6	9·05	77·19	7·80	—	5·96
Scotch (Heywood) cannel	16·8	53·94	—	—	—	46·06
Scotch (Lesmahagow) cannel	55·7	84·55	—	—	C_3H_8 ·91	14·54
Whitehill cannel shale	55·7	68·75	—	2·67	—	28·58
Whitby jet	30·2	10·93	—	—	86·90	2·17

AVERAGE COMPOSITION OF COAL GAS.

(*Thorpe.*)

Hydrogen	45·58
Methane	34·90
Carbon Monoxide	6·64
Ethene	4·08
Quartene	2·38
Sulphuretted hydrogen	0·29
Nitrogen	2·46
Carbonic acid	3·67
	100·00

ANALYSIS OF GASES ISSUING FROM THE MOUTH OF THE BESSEMER CONVERTER.

(*Snelus, I. and S. Inst.*)

	Time after Commencement of Blow.						After addition of Spiegeleisen at Bochum Works.	
	2 mins.	4 mins.	6 mins.	10 mins.	12 mins.	14 mins.		
Carbonic acid	10·71	8·59	8·20	3·58	2·30	1·34	—	0·86
Carbonic oxide	nil.	3·95	4·52	19·59	29·30	31·11	82·6	78·55
Oxygen	0·92	—	—	—	—	—	—	1·32
Hydrogen	88·37	0·88	2·00	2·00	2·16	2·00	2·8	2·52
Nitrogen		86·58	85·28	74·83	66·24	65·55	14·3	16·38

COMPOSITION OF GASES IN BLOWHOLES OF STEEL INGOTS.

(*Stead, Clev. Inst., Eng.*)

	Steel containing C = 0·42 p. c. Si = 1·00 p. c. Mn = 1·08 p. c.	Steel containing C = 0·33 p. c. Si = 0·10 p. c. Mn. = 0·69 p. c.	Steel containing C = 0·17 p. c. Si = 0·09 p. c. Mn. = 0·89 p. c.
Hydrogen	67·10	86·62	87·21
Nitrogen	30·30	13·29	11·15
Carbonic oxide	2·60	0·32	1·64
Oxygen	—	0·37	—

COMPOSITION OF VARIOUS GASES.

(By Weight.)

	N	H	CO_2	CO	CH_4	C_2H_4	Authority.
Blast furnace, Scotch	48·20	0·90	21·70	29·24	—	—	Bell
Blast furnace, Askam	52·59	0·14	13·47	33·80	—	—	Crossley
Blast furnace, Cleveland	58·54	0·06	14·32	27·03	—	—	Stead
Producer gas, Siemens	64·50	—	6·95	24·92	0·89	2·73	Snelus
Producer gas, Siemens	63·22	0·65	8·71	25·97	1·45	—	,,
Producer gas, Wilson	61·70	0·90	6·91	29·58	0·91	—	Stead
Producer gas, Wilson	62·84	1·11	8·29	26·33	1·43	—	,,
Retort-made town gas	—	8·17	—	18·61	56·10	17·12	—

Oil Gas.

The following are some interesting results obtained by Dr. Macadam on experimenting with Pintsch's and Keith's apparatus—

BLUE PARAFFIN OIL USED.

	Pintsch's Apparatus.			Keith's Apparatus.		
	A	B	Average.	A	B	Average.
Specific gravity of oil .	877·6	878·2	877·9	874·1	877·6	875·9
Flashing point	296°	294°	295°	292°	286°	289°
Firing point	356°	352°	354°	348°	346°	347°
Gas per gallon, cubic feet	90·7	103·4	97	85	84·8	84·9
Illuminating power . .	62·5	59·1	60·8	63·2	59·5	61·4
Volume of oil flowing into each retort per hour	1·4	1·18	1·29	2·3	1·3	1·8
Gas per retort per hour .	126·8	122·5	124·6	197·5	111·9	154·7
Heavy hydrocarbons, per cent.	39·2	37·1	38·2	39·9	38·2	39·0
Gas per ton, cubic feet .	23,128	26,356	24,742	21,772	21,671	21,721

PART IV.

WATER.

It is intended in this section to deal with waters required to be used (1) for boilers, and (2) for drinking purposes.

I.—WATER FOR BOILER PURPOSES.

Waters largely impregnated with carbonates and sulphates of lime and magnesia, are highly objectionable for use in steam boilers, since these salts are deposited as a hard incrustation on the boiler, tubes, &c., causing great inconvenience and waste of fuel, and any considerable quantities of such salts, as chloride, sulphate and carbonate of soda, &c., present in waters, although not causing "hardness," give much trouble in the form of "priming." Waters containing a comparatively large proportion of calcareous and magnesian salts are termed "Hard," while those containing a small proportion are termed "Soft." These qualities are practically determined by the more or less "curd" (lime and magnesian soaps) that is produced, when the water is used with soap. The amount of mineral matter or incrustating substances present in a water, materially depends upon the geological character of the strata through which the water passes or percolates. The following table shows the quality of the water obtained from the various geological strata or formations of this country:—

Formations.	Thickness in Feet.	Quality of Water.
Chalk	645 to 1,000	Hard.
Upper Greensand	100 to 400	Rather Hard.
Lower Greensand	20 to 500	Soft and Good.
Purbeck and Portland Beds	0 to 60	Rather Hard.
Coral Rag and Grit	40	„ „
Great and Inferior Oolites	200 to 450	Hard.
Upper Lias Sands	20 to 200	Soft.
Marlstone or Middle Lias	30 to 250	Rather Hard.
New Red Sandstone	0 to 2,150	Soft or Variable.
Lower Permian Beds	Variable.	Soft.

Collection of Samples.—For collecting the water, the stoppered glass bottles known as "Winchester quarts," holding 2½ litres, are generally found convenient. One of these filled will be sufficient for an ordinary analysis, but when an exhaustive examination is proposed at least three will be required.

If the water is to be taken from a pump or tap, let a few gallons of the water be run off before collecting; then fill the bottle to the top to expel any gases, empty, wash out the bottle about half a dozen times with the water, and then fill to within about an inch from the stopper, and tie down.

When the water is obtained from a well, tank, or river, the clean bottle is plunged into the water, and the neck kept below the surface, care being taken that no scum, &c., gets into the bottle, and that no mud or sediment is disturbed.

When the water is intended to be examined as to its suitability for dietetic purposes, it should be kept in a cool, dark place previous to its analysis, which should take place within forty-eight hours after its collection, since on long keeping, any organic matter may be decomposed by fermentation, &c.

In judging of the suitability of a water for boilers, &c., the following points must be determined :—(1) Mechanically suspended matter; (2) Total solid matter in solution; (3) Hardness—total, permanent, and temporary; (4) Chlorine in chlorides; (5) Alkalinity; and (6) Qualitative idea of the amounts of sulphates, nitrates, lime, and magnesia present.

This will give a tolerably good idea of the nature and suitability of water for use in steam boilers, although sometimes a complete mineral analysis of a water is desirable.

Determination of Suspended Matter.—If on shaking up the sample of water a fair quantity of suspended matter is seen, filter off about 250 cc. of the sample through a weighed filter, wash with distilled water, dry at 130° C., cool and weigh. Increase of weight multiplied by 280 = grs. suspended matter per gallon.

Total Solid Matter in Solution.—Accurately weigh a porcelain or platinum dish of about 100 cc. capacity, and measure out 70 cc. of the filtered sample from a pipette into it. Place on a water bath, evaporate to dryness, remove to an air bath regulated to 130° C. for one hour, transfer to a desiccator, allow to cool, and quickly weigh. Place in the air bath for another hour at 130° C., cool and weigh as before. If the second weight is no less than that of the previous one, the increase in weight over the dish = total solid matter in 70 cc., and which multiplied by 1,000 gives grains per gallon of solid matter.

Determination of Hardness.—The degree of hardness of a water is determined by ascertaining the amount of *standard* soap solution necessary to form a permanent lather with a definite volume of the sample: the "harder" the water the more soap will it consume, owing to the formation of insoluble calcium, magnesium, &c., soaps ("curd"), brought about by the decomposition of the soda or potash soap added, by the salts of the alkaline earths present in the water. The hardness of a water is usually expressed in terms of carbonate of lime, or degrees Clark (*i.e.*, grains per gallon), Dr. Clark being the originator of the process; and is usually determined in terms of "temporary" and "permanent" hardness. By "temporary" is meant that hardness which can be eliminated by boiling the water, by which means the carbonic acid gas which holds the carbonates of lime and magnesia, &c., in solution is expelled, and the carbonates in consequence precipitated. If this boiled water be filtered, the hardness ("permanent") of the filtrate will be diminished in proportion to the amounts of carbonates filtered off. Carbonate of lime is not, however, completely precipitated by boiling, 2·5 grains per gallon being soluble in water free from carbonic acid. In order to ascertain the hardness, a *standard* "hard" water and a *standard* soap solution will be required.

Preparation of Standard "Hard Water."—Dissolve 1·11 grms. of pure fused calcium chloride in a little water, and dilute to 1,000 cc. at 15° C., or dissolve 1 grm. of pure carbonate of lime in 50 cc. of 5 E HCl, evaporate to dryness, dissolve in 50 cc. of water, and carefully neutralize any trace of free acid with 5 E AmHO. In either case each cc. of the solution will correspond, or be chemically equivalent, to ·001 grm. of carbonate of lime.

Preparation of Standard Soap Solution.—Castile soap, which is supposed to be made with soda and olive oil, is much used for standard soap solutions, but the writer has found that it is liable to considerable deterioration on keeping, especially in cold weather, owing to the deposition of stearate and palmitate of soda; and warming up the solution with a view to their re-solution makes it more prone to deposit a fresh crop of precipitate, and in consequence it is never safe to use the solution unless "*standardized*" previous to use, which necessitates undesirable factors. Sodic oleate, supplied by Hopkin and Williams, London, makes a standard soap solution which suffers very little change on keeping, and can be generally recommended for the purpose. About 13 grms. of it are dissolved in a mixture of 500 cc. of methylated alcohol and 500 cc. of water, and filtered if necessary. It now becomes necessary to standardize it, so that 1 cc. will be equivalent to ·001 grm. of carbonate of lime. In order to effect this, 12 cc. of the standard hard water are run into a 250 cc. stoppered bottle from a burette and diluted to 70 cc. A burette is now filled with the soap solution, which is run into the bottle 1 cc.

at a time, and the bottle vigorously shaken after each addition, until a point is reached where a persistent lather, lasting for at least five minutes, is obtained. Note the volume required. 12 cc. of hard water should require 13 cc. of soap solution (distilled water itself requiring 1 cc. to form a lather), but it will be a figure less than this, and therefore the soap solution is too strong, and will require diluting, so that 12 cc. of standard "hard" water will require 13 cc. of soap solution. An example of an actual preparation of a standard soap solution will make matters clear :—

13 grms. of sodic oleate were dissolved in a mixture of 500 cc. of methylated alcohol and 500 cc. of water, and filtered. On testing in the manner described, 12 cc. of the standard "hard" water, diluted to 70 cc., required 11·4 cc. of the soap solution to form a persistent lather.

Now, since 13 cc. should have been required, every 11·4 cc. of the soap solution left, requires diluting by $13 - 11{\cdot}4 = 1{\cdot}6$ cc. There were 960 cc. of the solution left, therefore $\frac{960}{11{\cdot}4} = 84{\cdot}2$, and $84{\cdot}2 \times 1{\cdot}6 = 134{\cdot}7$ cc. more of the mixture of alcohol and water to be added.

On adding this quantity, thoroughly mixing, and testing as before, 12 cc. of the standard hard water required exactly 13 cc. of the soap solution.

Determination of Total Hardness.—70 cc. of the clear sample are run into a 250 cc. stoppered bottle, and the standard soap solution run into it in the manner described above, until a lather capable of persisting for five minutes is produced. The number of cubic centimetres required, minus 1 cc. for the water itself, will give the hardness in degrees Clark, or in terms of chalk in grains per gallon. If the water should require more than 16 cc. of standard soap solution, 70 cc. of distilled water must be added, and the testing continued as before, in order that the lathering may take place uniformly. Or 35 cc. of the sample are taken, diluted to 70 cc., and tested as before; but the result must be multiplied by 2. If the water contains a fair proportion of magnesia salts, there will be a little difficulty in obtaining the right point, owing to the slowness with which magnesia salts decompose soap; an apparent persistent lather is formed, which on being allowed to stand a little while and again shaken up, will disappear; a little experience with magnesian hard waters will familiarize the operator with this peculiarity.

The Permanent Hardness.—250 cc. of the clear sample are poured into a 500 cc. flask, and boiled for one hour, the original volume being kept up by frequent additions of boiling distilled water free from CO_2; after which it is covered over with a watch glass and put into cold water for a quarter of an hour to cool. It is now quickly poured into a 250 cc. graduated stoppered flask, diluted if necessary to exactly 250 cc. at 15° C. with distilled water free from CO_2, well mixed, and filtered. 70 cc. of the well-mixed solution are now poured into the bottle, and the permanent hardness determined as described.

The Temporary Hardness.—The temporary hardness, or that hardness removed by boiling, is obtained by deducting the degree of permanent hardness from that of the total.

Estimation of Chlorine in Chlorides.—Chlorine is mostly present in water as common salt (NaCl), but it is sometimes present as potassic chloride (KCl), calcium chloride ($CaCl_2$), and magnesium chloride ($MgCl_2$), so that it is wise to express the chlorine as chlorides, which of course could be run out in terms of common salt. The chlorine in waters is usually determined volumetrically by a standard solution of argentic nitrate, using potassic chromate as indicator of the end of the reaction.

Preparation of Standard Silver Nitrate Solution.—Dissolve 4·79 grms. of pure nitrate of silver in a little distilled water, dilute to exactly 1,000 cc. at 15° C. and thoroughly mix. Each cc. of this solution will exactly precipitate ·001 grm. ot chloride as chloride of silver.

$$AgNO_3 + NaCl = AgCl + NaNO_3.$$

Method.—Measure out 70 cc. of the sample into a porcelain basin of about 100 cc. capacity, and add to it two drops of E potassic chromate solution. Fill a burette with the standard silver nitrate solution, and run this gradually into the sample, stirring until the last drop added turns the solution from yellow to yellowish-red, owing to the formation of silver chromate. The principle of the process depends upon the fact, that silver has a greater affinity for chlorine than for chromic acid, and chromate of silver in consequence is not permanently produced until all the chlorine is precipitated, although when the silver solution drops into the sample tinted with potassic chromate, the silver chromate is momentarily formed, but is decomposed on stirring the solution, by the chlorides in excess, thus:

$$Ag_2CrO_4 + 2\ NaCl = Na_2CrO_4 + 2\ AgCl.$$

The number of cubic centimetres required for 70 cc. minus 0·1 cc. taken up for colouring this quantity of distilled water will represent the chlorine in terms of grains per gallon, and this multiplied by $\frac{117}{71}$ will give the number of grains in terms of common salt (NaCl) per gallon.

Determination of Alkalinity.—By alkalinity is understood the power of neutralizing an acid, and by noting the amount of standard acid of known strength required to neutralize a definite volume of water, the degree of alkalinity can be ascertained.

Preparation of Standard $\frac{E}{10}$ H_2SO_4.—Run out from a burette 50 cc. of standard E H_2SO_4 into a graduated 500 cc. flask, dilute with distilled water to 500 cc. at 15° C., and thoroughly mix. 1 cc. of this solution will exactly neutralize ·005 grm. of carbonate of lime or ·0053 grm. of carbonate of soda.

Preparation of Cochineal Solution.—Grind about 5 grms. of cochineal in a mortar, brush into a 300 cc. beaker, pour on 200 cc of water and 50 cc. of methylated alcohol, and allow to digest with frequent stirring for a few hours at about 60° C. Filter and bottle.

Method.—Measure off 250 cc. of the sample into a 300 cc. flask, add 2 cc. of cochineal solution, and mix; if carbonates be present in the water a pink colour will be produced. Treat 250 cc. of distilled water in the same manner, when a yellow solution is produced, which will be of service in ascertaining by comparison the colour indicating the end of the reaction with the $\frac{E}{10}$ H_2SO_4 on the sample. Fill a burette with the standard $\frac{E}{10}$ H_2SO_4, run it into the sample gradually, stirring, until the last drop turns the solution from pink to the colour of the tinted distilled water, and note the volume of solution required. It is customary to express the alkalinity in terms of grains of carbonate of lime per gallon, and the number of cubic centimetres of $\frac{E}{10}$ H_2SO_4 required for 250 cc. of water, multiplied by ·005 and 280, will give the desired figure.

Method for the Complete Mineral Analysis of a Water.—*Estimation of Silica, Oxide of Iron aud Alumina, Lime and Magnesia.*—Acidulate 1,000 cc. of the filtered sample with 10 E HCl, evaporate to complete dryness in a platinum or porcelain dish, and heat the residue to about 130° C. for about an hour, to render silica insoluble. Allow the dish to cool, moisten residue with 5 cc. of 5 E HCl, add 50 cc. of water, and allow to digest until all that is soluble is dissolved; filter off the insoluble residue, which is silica, through a small ashless filter, wash with hot water till free from chlorides, dry, ignite in weighed crucible, cool, and weigh; increase in weight multiplied by 70 = grains per gallon of silica. The solution is now made slightly alkaline with 20 E AmHO, gently boiled until the smell of ammonia has nearly all gone off, the precipitated oxide of iron and alumina filtered through a small ashless filter, washed, dried, ignited and weighed. The proportion of oxide of iron and alumina in water is usually so small that they are estimated and expressed together, and sometimes with the silica in addition. The filtrate from the iron oxide, &c., which should be about 150 cc. in volume, is now made strongly alkaline with, say, 5 cc. of 20 E AmHO, and 5 cc. of 5 E AmCl are added to keep magnesia in solution. 10 cc. of $\frac{E}{2}$ ammonic oxalate are now added, the solution well stirred, and the precipitated oxalate of lime thus produced allowed to subside for a few hours, after which it is filtered off through an ashless filter, washed with water, dried in the water bath, transferred to a weighed platinum crucible, heated gently at first, and finally for ten minutes to the highest heat of the blow-lamp. The oxalate thus becomes converted into anhydrous calcium oxide (lime); cool in the desiccator, and weigh. Increase in weight multiplied by 70 = grains of combined lime per gallon, and this figure multiplied by 0·7143, gives grains of calcium per gallon.

The filtrate is now evaporated to about 80 cc., 5 cc. of 20 E AmHO and 6 cc. of E sodic phosphate solution added, well stirred, and allowed to stand in the cold for about twelve hours. The magnesia is thus precipitated as ammonio-phosphate. It is filtered off, and the filtrate used with the aid of a feather to transfer the last portions of precipitate sticking to the beaker, to the filter. This method is adopted to limit the amount of wash water used, in which the precipitate is somewhat soluble; the precipitate is finally washed with a minimum quantity of 10 E AmHO. The filtrate is measured, and ·001 grm. allowed for each 50 cc., due to its solvent action on the ammonio-phosphate. The precipitate is dried, carefully ignited to a bright red heat in a weighed platinum or porcelain crucible, cooled, and the resulting pyrophosphate of magnesia ($Mg_2P_2O_7$) weighed. After adding the amount that was dissolved by the ammoniacal solution, multiply by ·2162 and 70, and the figure obtained expresses grains per gallon of magnesium.

Estimation of Potash and Soda.—1,000 cc. of the sample are evaporated to about 50 cc., about 0·15 grm. of caustic baryta added the solution boiled for a little while, and the precipitated carbonates, hydrate of magnesia, &c., filtered off. 5 E Am_2CO_3 is now gradually added until no further precipitate of carbonates of baryta and lime is produced. After allowing to settle, they are separated by filtration, the filtrate being received in a weighed platinum dish.

The solution now contains the sodium and potassium in the form of chlorides, together with ammoniacal salts; it is acidulated with 10 E HCl, evaporated to dryness, and the residue cautiously heated with the naked flame of a Bunsen burner to expel ammoniacal salts, which are driven off as dense fumes. Great care must be taken that the heat applied does not exceed very dull redness, or the chlorides of the alkalies will suffer loss by volatilization. After fumes have ceased to come off, the dish containing now KCl + NaCl is cooled in a desiccator and weighed. If the proportion of K and Na present be required, proceed as follows:—Dissolve in 10 cc. of water, add 4 cc. of E. platinic chloride, evaporate on water bath to a pasty condition, digest with 10 cc. of methylated alcohol, decant off the clear liquid from the precipitate of double chloride of platinum and potassium ($2\ KCl.PtCl_4$) through a weighed filter, add 5 cc. more alcohol to the residue, digest, and decant as before, repeating the digestion a third and fourth time, finally transferring the precipitate to the filter with the aid of a feather and a stream of alcohol from a small wash bottle. The filter and its contents are now dried in a water oven and weighed between two tared watch glasses. The weight of the $2\ KCl.PtCl_4$ thus obtained, multiplied by 0·3070 = KCl; subtract this from the weight of KCl + NaCl as obtained above; remainder = NaCl. Then—

KCl × ·5244 × 70 = grains K per gallon,

and

NaCl × ·3934 × 70 = „ Na „ „

Estimation of Sulphates—1,000 cc. of the clear sample are acidulated with 10 cc. of 10 E HCl, evaporated to about 250 cc. in a porcelain basin, transferred to a 300 cc. beaker, heated to boiling, 10 cc. of E baric chloride solution added, well stirred, and allowed to stand twelve hours. The resulting baric sulphate is then filtered off, washed, dried, scraped into a weighed crucible, filterer ignited separately, residue added to main portion, the whole ignited at a dull red heat for ten minutes, cooled and weighed. $BaSO_4 \times \cdot412 \times 70 =$ grains combined SO_4 (sulphion) per gallon.

Estimation of Nitrates.—A very neat method ot estimating the proportion of nitrates and nitrites in waters, is the adoption of Crum's method for the refraction of nitre, in which process the determination is deduced from the volume of nitric oxide gas given off when a concentrated solution of the sample is shaken up with 36 E H_2SO_4 and mercury, in a graduated tube termed a nitrometer. The following is the manner in which the process is carried out:—500 cc., or more if necessary, of the sample, are evaporated to dryness in a porcelain basin, and the residue drenched repeatedly with small quantities of hot water, the solution of the nitrates being assisted by rubbing the residue with the clean finger. Filter into a small beaker, and wash the residue with a little hot water to ensure complete solution of all the soluble nitrate. The solution is now to be evaporated to a volume not exceeding 1½ cc., and poured into the cup, A, of the nitrometer, Fig. 48, already filled with mercury. By lowering the tube D, and opening the tap B, the liquid is made to run into the graduated tube C, and by careful manipulation no air need enter; wash out the beaker with small quantities of water into C (using 3 cc. in all), and then pour 6 cc. of 36 E H_2SO_4 free from nitrates into the beaker. finally transferring it in small quantities at a time into C through B. Any air or CO_2 which is produced is now got rid of by rising D and carefully opening B. The tube, C, is now taken out of its clip, held at an angle of about 40°, and shaken in this plane

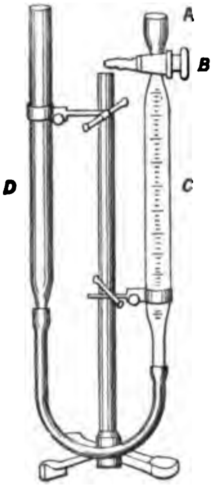


FIG. 48.

for about five minutes, part of the mercury being thus finely divided and mixed with the liquid. Allow to stand for five minutes, and again shake as before, until no further increase in the volume of gas given off takes place. Raise or lower D, as the case may be, until the level of the mercury therein is about one-seventh of the volume of the liquid above the level of the mercury in C. Read off the volume of nitric oxide (NO), and note the temperature and barometric pressure at the time.

Now since nitric oxide contains half its volume of nitrogen, the volume of gas thus found represents the volume of nitrogen from 1000 cc. of sample; 1000 cc. of nitrogen at 0° and 760 mm. mercury weigh 1·2544 grms., so that

$$\frac{\text{cc. of N (at } 0^\circ \text{ C. and } 760 \text{ mm.)} \times 1{\cdot}2544 \times 70}{1{,}000} =$$

grains of nitrogen per gallon, and the figure thus obtained multiplied by $\frac{62}{14}$ = grains NO_3 per gallon.

Estimation of Organic Matter.—The accurate determination of total organic matter in a water is attended with difficulties, owing to the decomposition of carbonate, nitrate, magnesic chloride, &c., during the ignition of the residue. However, a fair idea may be had of the proportion present by the following method:—Evaporate 500 cc. of the water to dryness in a platinum dish, and dry the residue at 130° C. until the weight remains constant; we thus obtain the total solid residue. It next becomes necessary to gently ignite so as to burn off all organic matter at as low a temperature as possible. For this purpose the dish is placed on a pipeclay triangle, fixed to a ring on a retort stand, and a flame, from a Bunsen burner, held in the hand, is made to play on the dish for ten minutes, care being taken that the temperature does not exceed a dull red heat. It is now cooled in a desiccator and weighed, and the ignition repeated as before until no further loss occurs. The CO_2 that has been driven off from the carbonates is now replaced by evaporating with strong CO_2 water, prepared by acting upon marble with 2 E HCl, washing the gas with water, and saturating distilled water with the washed gas. The dish is filled with CO_2 water thus prepared, and evaporated to dryness, the process being repeated four times more, which will be generally sufficient to replace all CO_2 lost by ignition. The residue is next dried at 130° C. until the weight remains constant. By subtracting this weight from that of the total solid residue the remainder is taken as organic matter.

Conversion of Solid Residue into Sulphates.—Often it is sufficiently accurate to determine the amount of combined sodium (disregarding potassium) indirectly. For this purpose the residue from the determination of the organic matter is evaporated to dryness with excess of E sulphuric acid to convert into sulphates, the residue gently ignited with ammonic carbonate, cooled, and weighed. The following analytical data of a complete analysis of a sample of water from the river Wye, at Ross, Monmouthshire, by the writer, illustrate the manner in which the bases and acids are combined, the combined sodium being in this case determined indirectly.

RIVER WYE WATER.

Estimation of Silica, Oxide of Iron, and Alumina.

1,000 cc. taken.

Porcelain Crucible marked 4	8·2225
Do. + SiO_2 + Fe_2O_3 + Al_2O_3	8·2262
	·0037

$·0037 \times 70 = 0·259$ grain per gallon.

Estimation of Combined Calcium.

1,000 cc. taken.

Porcelain Crucible, marked 1	6·4129
Do. + CaO + filter ash No. 3	6·4450
" " " 2nd ignition	6·4449
CaO + F.A.	·0320
F.A.	·0017
CaO	·0303

$·0303 \times ·7143 \times 70 = 1·52$ grains Ca per gallon.

Estimation of Combined Magnesium.

1,000 cc. taken.

Porcelain Crucible, marked 2	6·1902
Do. + $Mg_2P_2O_7$	6·2052
,, ,, 2nd ignition	6·2052
	·0150
Allowing for solubility of precipitate in ammoniacal solution	·0026
$Mg_2P_2O_7$	·0176

·0176 × ·2162 × 70 = 0·27 grain Mg. per gallon.

Estimation of Sulphates (SO_4).

1,000 cc. taken.

Porcelain Crucible, marked B	6·4129
Do. + $BaSO_4$ + F.A. No. 2	6·4412
,, ,, ,, ,, 2nd ignition	6·4412
	·0283
F.A.	·0009
	·0274

·0274 × ·412 × 70 = 0·79 grain SO_4 per gallon.

Estimation of Chlorine in Chlorides.

100 cc. taken.

1 cc. standard $AgNO_3$ = ·001 grm. Cl.
100 cc. sample required ·95 cc. standard $AgNO_3$.
·95 × ·7 = ·67 grain chlorine per gallon.
or $\frac{\cdot 67 \times 117}{71}$ = 1·1 grain common salt (NaCl) per gallon.

Total Hardness.

70 cc. of the sample required 6 cc. of standard soap solution to produce a persistent lather for 5 mins. 6 — 1 = 5° Clark.

Estimation of Nitrates.

500 cc. taken.

NO liberated	0·2 cc.
Temperature	19° C.
Barometric pressure	29·25″.

$$V = \frac{\cdot 2 \times 273 \times 29\cdot 25}{(273 + 19)\ 30} = \cdot 182 \text{ cc. at } 0^\circ \text{ and } 760.$$

Now a litre of nitrogen at 0° and 760 mm. weighs 1·2544 grms.

$$\frac{\cdot 182 \times 1\cdot 2544}{1000} = \cdot 000228 \text{ grm. N.}$$

$$\cdot 00023 \times 70 = \cdot 0161 \text{ grain N per gallon,}$$

or

$$\frac{\cdot 0161 \times 62}{14} = \cdot 0713 \text{ grain } NO_3 \text{ per gallon}$$

Alkalinity.

1 cc. standard $\frac{E}{10}$ H_2SO_4 = ·005 grm. $CaCO_3$

250 cc. of sample required 2·7 cc. of $\frac{E}{10}$ H_2SO_4 for neutralization.

2·7 × ·005 × 280 = 3·78 grains per gallon, expressed as carbonate of lime.

Estimation of Organic Matter.

500 cc. taken.

	Grms.
Platinum Dish	40·7432
Do. + residue dried for 2 hours at 130° C. . .	40·7946
,, ,, 1 ,, . .	40·7944
,, ,, ignited 10 mins. dull red heat .	40·7843
,, ,, ,, 2nd 10 mins. dull red heat .	40·7818
,, ,, ,, 3rd ,, ,, ,,	40·7804
,, ,, ,, 4th ,, ,, ,,	40·7800
After evaporating four times with CO_2 water and dried at 130° C.	40·7924
After evaporating once more with CO_2 water and dried at 130° C.	40·7925
Organic matter . .	·0019

·0019 × 2 × 70 = 0·27 grain organic matter per gallon.

Conversion of Solids into Sulphates.

500 cc. taken.

Platinum dish + sulphates, after ignition with am. carb. .	40·8010
,, ,, after 2nd ignition with am. carb.	40·8008
,, ,, ,, 3rd ,, ,, ,,	40·8008
,, ...	40·7432
	·0576

0576 × 2 × 70 = 8·06 grains per gallon.

Tabulated Results.

	Grains per gall.
Organic matter	0·27
Inorganic matter	6·90
Total Sulphate formed	8·06

Calcium	1·52
Magnesium	0·27
Silica, Oxide of Iron, and Alumina	0·26
Sulphion (SO_4)	0·79
Chlorine	0·67
Sodium (by calculation)	0·41
Nitrion (NO_3)	0·07
Carbonates (CO_3)	2·41
	6·40

and these may be combined as follows :—

$\frac{1·52 + 17}{5} = 5·17$ $CaSO_4$.
·27 × 5 = 1·35 $MgSO_4$.
$CaSO_4 + MgSO_4 + SiO_2$, &c. = 6·78.
8·06 — 6·78 = 1·28 Na_2SO_4.
71 : 23 : : 1·28 : x = ·41 Na.

46 : 117 : : ·41 : x = 1·04 NaCl.
1·04 — ·41 = ·63 combined Cl used
·67 — ·63 = ·04 Cl remaining.

71 : 111 : : ·04 : x = ·06 $CaCl_2$.
·06 — ·04 = ·02 Ca used.
1·52 — ·02 = 1·5 Ca remaining.

31 : 41 : : ·07 : x — = ·09 Ca $(NO_3)_2$.
·09 — ·07 = ·02 Ca used.
1·5 — ·02 = 1·48 Ca remaining.

12 : 17 : : ·79 : x = 1·12 $CaSO_4$.
1·12 — ·79 = ·33 Ca used.
1·48 — ·33 = 1·15 Ca remaining.

2 : 5 : : 1·15 : x = 2·88 $CaCO_3$.
2 : 7 : : 0·27 : x = ·95 $MgCO_3$.

Summary.

	Grains per gall.
Sodic Chloride	1·04
Calcic Chloride	0·06
Calcic Nitrate	0·09
Calcic Sulphate	1·12
Calcic Carbonate	2·88
Magnesium Carbonate	0·95
Silica, Oxide of Iron, and Alumina	0·26
Organic Matter	0·27
	6·67

Remarks.—Solids gave a slight odour of peat on ignition. Colour (before and after filtration) brownish-yellow. Suspended matter very small. Reaction : very slightly alkaline.

Owing to the small proportion of incrustating salts present, this water is of first class quality for use in steam boilers.

The following ingredients were obtained by Stillman in a sample of water by the method described on the opposite page :—

	Grains per Imperial gallon.
Silica	0·5740
SO_3	1·4420
Cl	0·3850
K_2O	0·0350
Na_2O	0·4340
MgO	0·5390
CaO	1·3580
$Fe_2O_3Al_2O_3$	0·3570
Organic	1·3510
Carbonic Acid	0·9590
	7·4340
Oxygen in excess of Cl	0·1120
	7·3220

Having determined the component parts of the water residue in grains per gallon, it becomes necessary to unite these in chemical union, as near as possible, as they exist in the water.

The general rule may be stated as follows :—The chlorine is combined with the sodium ; if still in excess, with the potassium, then magnesium, and finally calcium. The sulphuric acid is combined to the alkalies, provided there is not enough

THE FOLLOWING IS A TABULAR SCHEME FOR WATER ANALYSIS, ARRANGED BY PROFESSOR T. B. STILLMAN, OF NEW YORK.*

Evaporate two litres of the water in a weighed platinum capsule, upon a water bath, to dryness; transfer to a hot-air bath and heat at 105° C for thirty minutes; cool and weigh. Ignite slowly to a dull red heat until all carbonaceous matter is consumed; cool and weigh. The loss of weight equals organic and volatile matter. Warm the contents of the capsule with 10 cc. to 15 cc. of 10 E HCl, add 25 cc. H_2O, boil, and filter through an ashless filter into a 100 cc. graduated flask; wash thoroughly, make contents of flask to containing mark with H_2O; mix well.

<table>
<tr><td colspan="3">1. <i>Residue:</i>—Consists of insoluble mineral matter—SiO_2 or $SiO_2Al_2O_3$ ($CaSO_4$). It should be dried, ignited, and weighed; then fused with Na_2CO_3 in platinum crucible, dissolved in H_2O, made acid with HCl, evaporated to dryness with HCl, taken up with $H_2O + HCl$, and filtered.</td><td colspan="6">2. <i>Solution:</i>—100 cc.: Divide into two portions, one of 75 cc. for bases, and one of 25 cc. for SO_3. 75 cc.: Make alkaline with NH_4HO, boil, and filter (all weights obtained to be divided by 3 and multiplied by 4).</td><td rowspan="5">CO_2:
Is found by combining the chlorine and sulphuric acid with the bases, then determining how much, CO_2 is required to convert the rest of the CaO and MgO to carbonates, as shown in the example given below.</td><td rowspan="5">Cl: Concentrate 250 cc. of the water in a porcelain dish to about 50 cc.; add a few drops of K_2CrO_4 sol., and titrate with a standard solution of $AgNO_3$, in which each cc. corresponds to 0·0017 grm. chlorine. Multiply result by 4 = grms. of Cl per litre.</td></tr>
<tr><td rowspan="4"><i>Residue:</i>—SiO_2; ignite and weigh as SiO_2.</td><td colspan="2"><i>Filtrate:</i>—Make alkaline with NH_4HO, boil, and filter.</td><td rowspan="4">3. <i>Residue:</i>—Consists of $Al_2O_3Fe_2O_3$. Dry, ignite, and weigh as such.</td><td colspan="4">4. <i>Filtrate:</i>—Add solution of $NH_4\bar{O}$; set aside 3 hours, then filter.</td><td rowspan="4">25 cc.: Warm, add solution of barium chloride, and allow to settle 3 hours; filter, wash, dry, ignite, and weigh as $BaSO_4$. Calculate to SO_3, and multiply result by 4.</td></tr>
<tr><td rowspan="3"><i>Residue:</i>—$Al_2O_3Fe_2O_3$, dry, ignite, and weigh as such.</td><td rowspan="3"><i>Filtrate:</i>—Add solution of $NH_4\bar{O}$, set aside 3 hours, filter, dry, ignite, and weigh as CaO. Calculate to $CaSO_4$.</td><td rowspan="3">5. <i>Residue:</i> Consists of $Ca\bar{O}$. Dry, ignite, and weigh as CaO.</td><td colspan="3">6. <i>Filtrate:</i>—Evaporate to dryness in platinum dish; ignite to expel all ammonia salts; cool, add water, boil, and filter; wash well.</td></tr>
<tr><td rowspan="2">7. <i>Residue:</i> Consists of MgO, HO. Dry, ignite, and weigh as MgO.</td><td colspan="2">8. <i>Filtrate:</i>—Transfer to a weighed platinum dish, add a few drops of H_2SO_4. evaporate to dryness, and ignite to constant weight. This residue is composed of Na_2SO_4, $MgSO_4$, (K_2SO_4); after weighing, dissolve in H_2O, make solution up to 50 cc.; mix thoroughly, and divide into two equal portions of 25 cc. each.</td></tr>
<tr><td>1st—25 cc.: Add a few drops of HCl make alkaline with NH_4HO; then add, with constant stirring, solution of Na_2HPO_4; set aside 3 hours, filter, dry, ignite, and weigh as $Mg_2P_2O_7$; calculate this to $MgSO_4$, and subtract, after multiplying by 2, from total weight of sulphates in (8), and then the $MgSO_4$ is calculated to MgO.</td><td>2nd—25 cc.: Add a few drops of HCl, then solution of Pt_2Cl_4; evap. on water-bath with some alcohol. Filter off the K_2PtCl_6 on small tared filters, dry, and weigh as K_2PtCl_6. Calc. this wt. to K_2SO_4 and subtract, after multiplying by 2, from wt. of Na_2SO_4 and K_2SO_4, and the residue left will be the weight of Na_2SO_2. The K_2SO_4 and Na_2SO_4 are then calculated to K_2O and Na_2O.</td></tr>
<tr><td>SiO_2.</td><td>$Al_2O_3.Fe_2O_3$</td><td>(Insol.)
$CaSO_4$.</td><td>(Sol.)
$Al_2O_3.Fe_2O_3$</td><td>CaO.</td><td>MgO.</td><td colspan="2"></td><td>SO_3.</td><td>CO_2.</td><td>Cl.</td></tr>
<tr><td>+</td><td>+</td><td>+</td><td>+</td><td>+</td><td>+</td><td>+</td><td>+</td><td>+</td><td>+</td><td>+</td></tr>
</table>

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* *Steven's Indicator*, vol. 7, No. 4.

chlorine to saturate them; then to the calcium, and finally to the magnesium.

The carbonic acid is united with the calcium and magnesium after the other combinations are made. There are exceptions to this rule; mineral waters and many artesian well waters forming notable examples.

Carrying out the above, the following is obtained:—

	Grms. per litre.	Grains per Imperial gallon.
$NaCl$	0·0091	0·6370
Na_2SO_4	0·0033	0·2310
K_2SO_4	0·0009	0·0630
$CaSO_4$	0·0311	2·1770
$CaCO_3$	0.0118	0·8260
$MgCO_3$	0·0162	1·1340
$Fe_2O_3Al_2O_3$	0·0051	0·3570
SiO_2	0·0082	0·5740
Organic, &c.	0·0193	1·3510
Total	0·1050	7·3500

This analysis shows, that the principal scale-forming ingredient is calcium sulphate, being more than equal to the carbonates of calcium and magnesium.

The following analysis is of a water containing sulphuric acid, but the alkalies being present in sufficient amount to combine with all of it, as well as the chlorine, no sulphate of lime is present.

	Grms. per litre.	Grains per Imperial gallon.
SiO_2	0·0038	0·2660
SO_3	0·0110	0·7700
Cl	0·0062	0·4340
K_2O	0·0033	0·2310
Na_2O	0·0185	1·2950
MgO	0·0165	1·1550
CaO	0·0466	3·2620
$Al_2O_3Fe_2O_3$	0·0020	0·1400
Organic Matter	0·0246	1·7220
Carbonic acid	0·0530	3·7100
	0·1855	12·9850
Oxygen in excess of Cl	0·0021	0·1470
Total	0·1834	12·8380

Combined as follows :—

	Grms. per litre.	Grains per Imperial gallon.
$NaCl$	0·0154	1·0780
Na_2SO_4	0·0141	0·9870
K_2SO_4	0·0061	0·4270
$CaCO_3$	0·0833	5·8310
$MgCo_3$	0·0338	2·3660
$Al_2O_3Fe_2O_3$	0·0020	0·1400
SiO_2	0·0038	0·2660
Organic	0·0246	1·7220
Total	0·1831	12·8170

Where all the chlorine is not in combination with the sodium and potassium, chloride of magnesium is usually present.

This latter compound, while not scale-forming, is considered an active corrosive agent—upon the supposition that at the temperature of 100° C., and higher, it is decomposed, and hydrochloric acid formed and liberated.

The report, given below, is of a water from a driven well in Florida. Complaint having been made that not only was the scale excessive in amount, but that corrosive action was also very marked, an analysis was made, reference to which readily explains the difficulty encountered in the boilers.

	Grms. per litre.	Grains per Imperial gallon.
$NaCl$	0·323	22·61
KCl	0·067	4·67
$MgCl_2$	0·104	7·28
$CaSO_4$	0·197	13·79
$CaCO_3$	0·293	20·51
$MgCO_3$	0·144	10·08
SiO_2	0·011	0·7
$Al_2O_3Fe_2O_3$	0·007	0·49
Organic	0·138	9·66
Total	1·284	89·88

In all of the above analyses the constituents have been stated in grains per British imperial gallon, rather than in parts per 100,000, the former being in general use by the mechanical profession as the proper method by which to express the weights of the component parts of water.

Analyses of River Waters.—The following are analyses of various river waters, expressed in parts per 100,000 :—

The Thames at Twickenham.

Total solid contents 32·01.

Carbonate of Lime	18·23	Sulphate of Potash	0·95
„ Magnesia	1.47	Chloride of Lime	2·50
Sulphate of Lime	0·64	Silica	0·39
„ Soda	2·86	Organic Matter	4·97

The Seine above Paris.

Total solid contents 17·90.

Carbonate of Lime	9·20	Sulphates of Soda and Magnesia	1·00
„ Magnesia	3·90	Chloride of Lime	1·00
Sulphate of Lime	2·00	Silica, Alumina, Iron Oxide	0·80

The Rhine at Strassburg.

Total solid contents, 23·18.

Carbonate of Lime	13·56	Nitrate of Potash	0·38
„ Magnesia	0·51	Silica	4·88
Sulphate of Lime	1·47	Alumina	0·25
„ Soda	1·35	Iron Oxide	0·58
Chloride of Soda	0·20		

The Rhone at Geneva.

Total solid contents 18·20.

Carbonate of Lime	7·89	Chloride of Soda	0·17
„ Magnesia	0·49	Nitrate of Soda	0·85
Sulphate of Lime	4·66	Silica	2·38
„ Magnesia	0·63	Alumina	0·39
Sulphate of Soda	0·74		

THE DANUBE AT VIENNA

Total solid contents 12·62.

Carbonate of Lime	8·37	Sulphate of Potash and Soda	0·20
,, Magnesia	1·50	Silica	0·49
Sulphate of Lime	0·29	Iron Oxide	0·20
Sulphate of Magnesia	1·57		

THE SPREE AT BERLIN.

Total solid contents 11·40.

Carbonate of Lime	6·50	Chloride of Soda	1·20
,, Magnesia	0·90	Nitrate of Soda	0·30
Sulphate of Soda	0·60	Alumina and Iron	1·30
Sulphate of Potash	0·60		

The following are analyses by Professor Wanklyn, of waters supplied to the Metropolis by three of the principal water companies :—

WEST MIDDLESEX COMPANY.

(Grains per gallon).

Silica	0·3	Sulphate of Magnesia	0·6
Carbonate of Lime	12·9	Nitrate of Magnesia	1·1
Sulphate of Lime	2·4	Chloride of Sodium	2·0

NEW RIVER COMPANY.

Silica	0·26	Nitrate of Lime	1·00
Alumina, &c.	0·14	,, Magnesia	1·28
Carbonate of Lime	12·70	Chloride of Sodium	2·02
Sulphate of Lime	1·60		

KENT COMPANY.

Silica	0·75	= 18·3 insoluble solids.
Alumina, &c.	0·22	
Carbonate of Lime	16·30	
Water	1·03	

KENT COMPANY (*continued*).

Silica, Alumina, &c.	0·28	
Sulphate of Lime	5·37	
„ Magnesia	0·93	
Nitrate of Magnesia	1·20	= 12·0 soluble solids.
„ Soda	1·21	
Chloride of Soda	2·64	
Water	0·37	

DEEP WELL WATER AT CROYDON, SURREY.

(Grains per gallon).

Total solid contents	21·6	Sulphate of magnesia	1·4
Silica	trace	Chloride of soda	1·8
Carbonate of lime	14·1	Nitrate of soda	1·4
Sulphate of lime	1·8	„ potash	1·1

Sea Waters.—The following are analyses of various sea waters, from which it is seen that sodic chloride is present to the extent of nearly 3 per cent., the other constituents being comparatively small.

	British Channel. (Schweitzer). Pts. per 1000.	Irish Sea. (Thorpe). Pts. per 1000.	Mediterranean. (Usiglio). Pts. per 1000.
Water	963·74372	966·14054	962·345
Sodic Chloride	28·05948	26·43918	29·424
Potassic Chloride	0·76552	0·74619	0·505
Magnesic Chloride	3·66658	3·15083	3·219
Magnesic Bromide	0·02929	0·07052	0·556
Magnesic Sulphate	2·29578	2·06608	2·477
Calcic Sulphate	1·40662	1·33158	1·357
Calcic Carbonate	0·03301	0·04754	0·114
Iodine	traces	—	—
Ammonic Chloride	„	0·00044	—
Ferrous Carbonate	—	0·00503	0·003
Magnesic Nitrate	—	0·00207	—
	1000·00000	1000·00000	1000·000
Specific Gravity	1027·4 at 16°	1024·84 at 15°	1025·8 at 21°

According to Dittmar the solids obtained from sea water have the following percentage composition :—

Chloride of Sodium	77·76
Chloride of Magnesium	10·88
Sulphate of Magnesia	4·74
Sulphate of Lime	3·60
Sulphate of Potash	2·46
Bromide of Magnesium	0·22
Carbonate of Lime	0·34
	100·00

The Softening of Hard Waters.—Of late years considerable attention has been paid to the question of softening of hard waters intended for use in steam boilers. The importance of obtaining soft waters for boilers cannot be too strongly urged upon steam-users, especially from an economic point of view. The loss of heat, and consequent increase in the consumption of fuel, attending the deposition of incrustating salts on boiler tubes, &c., is very great, and, owing to the incrustation being such a bad conductor of heat, the wear and tear of a boiler is materially increased by augmenting unequal expansion, due to over-heating of tubes, &c. It is computed that $\frac{1}{16}$ inch thick of incrustation in a boiler increases the consumption of fuel by about 15 per cent. in the production of a given quantity of steam, while a layer of $\frac{1}{4}$ inch causes a dead loss of 30 per cent. of fuel.

The number of chemical substances which have been used, and suggested, to be placed in boilers to remedy the evils brought about by the use of hard waters is legion. The following are a few of the substances that have been used, and said to prevent or remove incrustations :—Tannate of soda, caustic soda, tannic acid, molasses, potatoes, Irish moss, petroleum, tallow, spoiled bacon, ammonium chloride, Barium chloride, besides many patent "fluids" and "compositions." The introduction of oil or grease into a boiler prevents, to a great extent, the cementation of the precipitated carbonates and sulphates on to the tubes, but unless frequent blowing-off is resorted to serious consequences may accrue, due to a combination of the carbonates of lime and magnesia with the oil; explosions are said to have been brought about by this cause.

The writer's experience has shown that the successful and economic treatment of hard water must be performed prior to its entering the boiler. Many of the nostrums used while remedying one evil produce others, such as increasing the soluble matter of the water and causing priming, corrosive action on tubes and plates, &c.

The most economic process for softening hard waters, at least those waters containing the carbonate of lime and magnesia in solution, is undoubtedly that devised by the late Dr. Clark. The principle of the process depends upon the fact that the carbonates of lime and magnesia are held in solution by an excess of carbonic acid, and if the excess of carbonic acid be removed by boiling or neutralization, the carbonates are no longer soluble in the water and are at once precipitated. In Dr. Clark's process a clear solution of lime—prepared by slaking quick-lime and adding a large quantity of water, allowing any excess of lime hydrate to subside—is added to the water intended to be softened until the whole of the free carbonic acid is neutralized. The free carbonic acid in the hard water combines with the lime in the lime water with the formation of carbonate of lime, which is co-precipitated with the carbonates of lime and magnesia in the hard water, thus—

$$CO_2 + Ca(OH)_2 = Ca\,CO_3 + H_2O.$$

In judging if the right quantity of lime water has been added, a little of the water is taken out of the softening tank and poured into a test tube; a few drops of silver nitrate solution is then added—if there be no excess of lime a white turbidity is produced: a slight excess of lime produces a straw colour, and a greater excess will produce a brown precipitate.

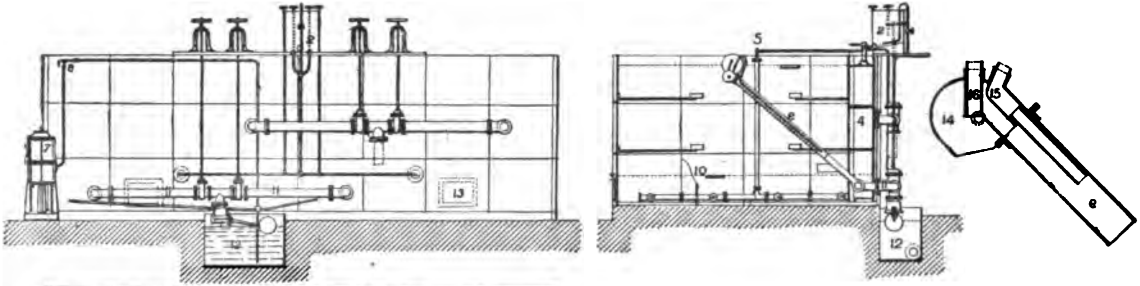
In Dr. Clark's original process, for which he took out a patent in 1841, no provision was made for filtering the precipitate carbonates; the softened water was to be allowed to clarify by subsidence, which necessitated the use of large and costly tanks, and the time required for complete subsidence was so long as to render the process practically useless where a continuous and large supply of softened water was required; and, moreover, the sulphates of lime and magnesia present in the water were not eliminated by the process. Since Dr. Clark's time there have been many ingenious arrangements for softening and rapid filtration of the precipitates. Mr. Porter appears to have been the first to invent a patent filter for the continuous softening and filtration of water. Many others have likewise made improvements in the mechanical details essential to rapid filtration. Atkins, Gaillet and Huet, Howatson, Maignen, and, more recently, Archbutt and Deeley, have invented processes and mechanical arrangements, which now make the question of water-softening easy, economic, and efficacious.

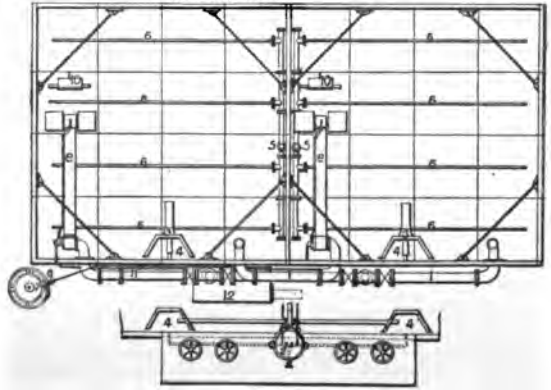
It will not be necessary for the purpose of this work to go into the merits and demerits of all the methods of softening and filtration, but a description of the Archbutt and Deeley process may be given, as being, in the writer's opinion, one of the best of modern processes. The author is indebted to Mr. Archbutt for a communication on the subject.

Figs. 49 and 50 show arrangements for the treatment of 5,000 to 10,000 gallons of water per hour. The reservoir for softening the hard water is a cast iron tank 32 ft. by 16 ft. and 10 ft. high, divided into two equal parts which are used alternately, thus ensuring a continuous supply of softened water. The hard water is run into either tank through the pipe (1), which is attached to the main, until it is on a level with the top of the well (4).

In this process the chemicals used are lime and soda ash, which soften water of temporary and permanent hardness: definite quantities of these chemicals together with in some cases a small quantity of sulphate of aluminium (about 1½ ozs. per 1,000 gallons) are dissolved in hot water in the small tank (2). The liquor is then run into the well, the trajector (3) at the same time being put into action by opening the steam valve, thus causing a rapid current of water producing a

FIG. 49.





LIST OF REFERENCES.

1 Hard water supply.
2 Tank for dissolving chemicals.
3 Trajector } for mixing.
4 Well
5 Blower for air } for stirring mud.
6 Perforated pipes
7 Coke stove.
8 Blower and pipe for fuel gas.
9 Floating discharge and carbonating pipe.
10 Rest with pivoted baffle plate for discharge pipe.
11 Fixed discharge pipe.
12 Tank with ball tap for supply of purified water.
13 Mud door.
14 Discharge nozzle.
15 Gas inlet.
16 Gas escape.

FIG. 50.
PATENT HARD WATER PURIFIER, ARCHBUTT AND DEELEY'S INTERMITTENT SYSTEM.

thorough mixing of the hard water and chemicals. After the mixture has stood for a short time a little of the softened water is taken out and a few drops of nitrate of silver added, to test for free lime; any excess of lime will cause a brown discoloration, while if the proper quantity of lime has been added a light straw-colour is produced. It has been found that if the mud produced on softening a water is stirred up with the water rapid subsidence afterwards takes place, due no doubt to the adhesion of the more finely divided precipitate to the coarser particles present. In order to bring about this end steam is admitted to the blower (5), which carries down air with it, and which is forced out of the perforated tubes fixed at the bottom of the tank; the currents of air as they rise in the tank cause a quantity of the sludge to diffuse through the liquid; after the steam has been on for about a quarter of an hour, the sediment is allowed to subside for about half an hour, when it will be found that about 6 feet of the water from the top will be so clear as to contain not more than about 1 grain per gallon of suspended matter. When there is much magnesia salt present in the water there would appear to be formed in the softened a small quantity of hydrate of magnesia, which though trifling in amount causes much trouble in being deposited in hot injectors, feed pipes, and clack boxes. The cause of the presence of hydrate of magnesia is due to the action of the lime water and carbonic acid upon any carbonate of magnesia, thus :—

$$\mathrm{Mg\,CO_3 + CO_2 + 2\,Ca(OH)_2 = 2\,CaCO_3 + Mg(OH)_2 + H_2O.}$$

To remedy the deposition it has been found that if the softened water is treated with carbonic acid no precipitation takes place on heating. Consequently there is an arrangement made for the absorption of CO_2 by the softened water, which is produced by the combustion of coke in the coke store (7); the gas produced is continually forced by means of the blower (8) along the horizontal limb of the gas-pipe down the vertical limb to the four-way junction, where there is an outlet for the condensed water into the small tank (12); the gas passes thence up the sloping limb through the side of the softening tank to a small swivel which works concentrically with the large swivel of the discharge-pipe; from the swivel the gas rises through a small pipe fixed to the discharge-pipe and finally enters the nozzle of the latter through the gas-inlet (15) shown in the enlarged section of the nozzle (14). On leaving the end of the inlet-pipe the gas is caught by the current of water flowing down the discharge, which is repeatedly splashed upwards by the ribs fixed at intervals along the bottom of the pipe, and in this way a thorough mingling of the gas and water is effected. The gas and water pass together through the ball-tap fixed over the small supply tank (12), into which the softened and carbonated water falls, and from which it is drawn off for use, whilst the residual nitrogen, &c., of the gas escapes into the air. If the small tank should fill up and cause the ball-tap to close the outlet of the discharge-pipe, the gas then accumulates in the pipe until the water in the nozzle is depressed below the orifice of the gas-escape (16), up which the gas passes into the atmosphere without bubbling through and disturbing the water in the softening tank.

When the water in the softening tank is getting low, the discharge nozzle, before it can approach too near the mud at the bottom of the tank, engages and tilts the baffle plate pivotted on the rest (10). This keeps the pipe from falling any lower, and also prevents the mud from being disturbed and drawn into the pipe. A strip of angle-iron fixed on the side of the tank two feet from the bottom indicates the level down to which the water is drawn off. When the mud has attained a thickness of about 12 inches it is stirred up and run out of the tank through the mud doors (13). About an inch of mud is left in the tank to carry on the clarifying process.

If a hard water contains calcium carbonate with but trifling amounts of other salts there would be no necessity to use carbonate of soda (58 per cent. soda ash), the cost of which is about seven times as much as lime.

When, however, sulphate of lime, and also magnesium salts are present, both lime and soda ash are required.

The following are reactions which take place when hard waters containing the carbonates and sulphates of lime and magnesia are treated with lime and soda ash :—

(1) $CaCO_3.CO_2 + Ca(OH)_2 = 2\ CaCO_3 + H_2O.$
(2) $MgCO_3 + CO_2 + 2\ Ca(OH_2) = 2\ CaCO_3 + Mg(OH)_2 + H_2O.$
(3) $CaSO_4 + Na_2CO_3 = CaCO_3 + Na_2SO_4.$
(4) $MgSO_4 + Ca(OH)_2 + Na_2CO_3 = CaCO_3 + Mg(OH)_2 + Na_2SO_4.$

With regard to the cost of softening waters, the harder the water the more chemicals will be required for softening, and therefore the greater will be the cost. Dr. Frankland states that to soften 700 gallons of water with lime only it is necessary to add 1 oz. of quicklime for each part of temporary hardness in 100,000 parts of water.

The following is an example of the manner in which the amount of lime required is calculated, together with its cost, to remove the temporary hardness from 1,000 gallons of water :—

A sample of water had 14·7 degrees of temporary hardness. 70,000 : 100,000 : : 14·7 : x = 21 parts hardness per 100,000 parts. 21 ozs. of lime is therefore required for 700 gallons, and

$$\frac{1{,}000 \times 21}{700} = 30 \text{ ozs.} = 1{\cdot}88 \text{ lbs. for } 1{,}000 \text{ gallons.}$$

Lime costs, say, £1 = 240 pence per ton of 2,240 lbs., then

$$\frac{1{\cdot}88 \times 240}{2{,}240} = 0{\cdot}201 \text{ pence per } 1{,}000 \text{ gallons.}$$

The following are analyses of eight samples of water, together with the cost of chemicals needed to reduce the hardness in each case to about 3½ degrees by the Archbutt and Deeley process :—

	Grains per gallon.							
	1	2	3	4	5	6	7	8
Calcium carbonate .	8·74	13·15	16·39	10·99	2·06	9·41	8·34	1·39
Magnesium Carbonate	2·78	0·33	0·31	2·76	0·94	1·00	2·82	1·78
Calcium sulphate .	3·26	—	4·30	2·99	47·34	22·91	40·61	54·14
Magnesium sulphate .	—	1·96	1·28	12·41	5·70	15·90	22·25	22·46
Sodium sulphate. .	1·44	0·30	—	18·96	9·98	—	2·65	28·96
Magnesium nitrate .	—	—	—	—	—	11·50	—	—
Sodium nitrate . .	—	0·96	traces	—	—	—	—	—
Magnesium chloride .	—	—	—	—	—	2·08	—	—
Sodium chloride. .	2·72	2·06	3·05	5·28	6·77	5·05	6·35	5·28
Silica, &c. . . .	0·43	0·39	0·42	0·31	0·62	0·90	0·84	0·36
Total solid matter	19·43	19·15	25·75	53·70	73·41	68·75	83·86	114·37
Total lime (CaO) .	6·24	7·36	10·95	7·39	20·64	14·70	21·39	23·07
Total magnesia (MgO)	1·33	0·81	0·58	5·48	2·36	9·82	8·81	8·38
Total hardness (= *Calcium carbonate equivalent to total lime and magnesia*) . . .	14·5	15·16	20·99	26·77	42·7	50·57	60·02	61·95
Cost of chemicals required for softening 1,000 gallons to about 3½ degrees lime computed at £1 per ton and 58°/₀ soda ash at £6 17s. 6d. per ton	¾d.	½d.	1d.	2d.	4¾d.	5d.	6d.	7d.

The following analysis shows the composition of the water used by the Midland Railway at Derby before and after treatment by the Archbutt and Deeley process:—

Before Treatment.

	Grains per gallon.	
Calcium carbonate	9·74	containing 7·78 grains lime.
Calcium sulphate	5·64	
Magnesium carbonate	2·45	containing 1·62 grains magnesia.
Magnesium sulphate	1·35	
Sodium sulphate	3·41	
Sodium chloride	2·81	
Silica	0·34	
Total solids	25·74	
Calculated hardness	17·9°	

After Treatment.

Calcium carbonate	2·25	containing 1·26 grains lime.
Magnesium carbonate	2·18	containing 1·04 ,, magnesia.
Sodium sulphate	11·33	
Sodium carbonate	0·85	
Sodium chloride	3·38	
Silica	0·48	
Total solids	20·47	
Calculated Hardness	4·8°	

II. WATER FOR DRINKING PURPOSES.

In judging of the suitability of a water for general domestic use, the following data will usually be found sufficient to form an opinion as to its quality:—Total Solids; Chlorine; Free and Albuminoid Ammonia; Nitrites and Nitrates; Hardness; Poisonous Metals; Colour before and after Filtration; Suspended matter; and The Odour of the Water when warmed. In some cases, such as the question of a town supply, a complete mineral and biological analysis should be made in addition.

The Total Solids.—These may be determined as directed on page 198. They should be gently ignited; and an observation of the odour given off will serve to show whether any organic matter present is of animal or vegetable origin. With regard to the amount of total solid matter present, which may allow the water to be pronounced fit for drinking, Dr. Wanklyn states that if it does not exceed 30 or 40 grains per gallon there would be no reason for rejecting it on that score.

The Chlorine.—The chlorine is to be determined as directed on page 201. The amount of chlorine present in a sample of water proposed to be used for drinking forms an important datum, since it is, within certain limits, an indication of sewage contamination, animal secretions being highly charged with salts in which chlorine is a heavy constituent, whereas natural waters usually contain but very small proportions of chlorine, often only one grain in the gallon. Although a water may contain a comparatively high proportion of chlorine, it does not *always* follow that it is charged with sewage since the geological strata through which the water passes may afford it an unusually high amount of chlorine. A water containing little or no chlorine is sure not to contain any sewage, but at the same time it may be charged with objectionable decomposing vegetable organic matter, so that the amount of chlorine present, as seemingly indicating presence or absence of organic matter, must be taken with some reservation. The amount of chlorine in sewage is from 9 to 12 grains per gallon.

Free and Albuminoid Ammonia.—The estimation of these bodies, known as Wanklyn's ammonia process, affords valuable criteria as to the purity of a water, organically considered. The "free" ammonia is that which can be removed by simple distillation of the water, while the "albuminoid" ammonia does not pre-exist as ammonia in waters, but is produced by the conversion of the nitrogen, contained in albuminous or nitrogenized organic matter present, by the action of a solution of potassium permanganate, made strongly alkaline with potassic hydrate. The whole of the nitrogen is not liberated from albuminoid substances as ammonia by this means, but the amount given off is a fraction of the total, being thus proportional to the quantity of nitrogenous or albuminoid substances present.

The ammonia process requires the following standard solutions, &c.:—

Dilute Standard Solution of Ammonia.—Two solutions are generally kept: a strong solution which contains 1 milligram of ammonia in each cubic centimetre, and a weak solution prepared by diluting 1 volume of the former to 100 volumes, which will thus contain ·01 of a milligram of ammonia in each cubic centimetre. The strong solution is prepared by dissolving 3·15 grms. of pure dry ammonic chloride in water, diluting to 1,000 cc. at 15° C., and well mixing; and to prepare the weak solution 10 cc. of the solution thus prepared are run from a burette into a graduated litre flask, and diluted to 1,000 cc. at 15° C.

Alkaline Potassic Permanganate Solution.—Weigh out 8 grms. of potassic permanganate and 200 grms. of stick potassic hydrate, into a 1½ litre basin, add 1,000 cc. of distilled water free from ammonia, and stir until all is gone into solution; the solution is now boiled, until about one quarter of the liquid has boiled off, diluted to original bulk, again boiled down as before, and finally diluted to 1,000 cc. and bottled. It is necessary to give the solution a thorough boiling in order to drive off any ammonia that may be present. Mere evaporation below the boiling point does not appear to be very effective in removing any traces of NH_3 present. 50 cc. of this solution are required for each analysis, and the amount of ammonia still left in this quantity must be determined by the method herein described, using water free from ammonia in the distillation. If any appreciable quantity of ammonia be thus found, it must be deducted from the amount obtained for albuminoid ammonia in each analysis.

The Nessler Reagent.—This extremely delicate test for ammonia is prepared as follows:—Weigh out 35 grms. of potassic iodide and 13 grms. of mercuric chloride (*corrosive sublimate*) in a 1½ litre beaker, dissolve in about 800 cc. of hot distilled water, and allow to cool; a saturated solution of mercuric chloride is now cautiously added until a slight but permanent precipitate of red periodide of mercury is produced, the solution being, of course, well stirred during the addition. 160 grms. of potassic hydrate or 120 grms. of sodic hydrate are now added to the liquid and stirred until it all goes into solution, which is finally to be diluted to 1,000 cc. The solution is now to be rendered sensitive by adding at first 2 cc. of saturated mercuric chloride solution, well shaken, and the precipitate allowed to settle. On adding 2 cc. of Nessler reagent to 50 cc. of water containing 0·5 cc. of the dilute ammonia solution, an immediate yellowish-brown colour should be produced, owing to the formation of dimercur-ammonium iodide NHg_2IOH_2. If this is not the case the solution is not sufficiently sensitive, and more mercuric chloride solution must be added until the desired delicacy is produced.

Distilled Water free from Ammonia.—It is very obvious that, in testing for such minute quantities of ammonia, the water used for diluting, &c., should be free from all traces of ammonia, or must not contain more than ·005 milligram in 100 cc. The writer prepares distilled water free from ammonia, by distilling good river water. The first 10 per cent. of distillate is rejected; the next 30 per cent. put into the ordinary stock bottle, the next 20 per cent. is bottled separately, and will be found tolerably free from ammonia. The rest of the distillate, which contains traces of ammonia, is put with the ordinary stock.

It is needless to remark that all apparatus must be scrupulously clean for this process, and that all bottles or any other vessel containing solutions from which ammonia is liable to be given off must be kept out of the room for the time the analysis is on, or locked in a cupboard some distance away, or placed in a good stink closet, provided with a good draught.

Estimation of Free Ammonia. — A stoppered litre glass retort is selected, and about 10 cc. of strong sulphuric acid poured into it, which is allowed to moisten the whole of the interior of the retort, after which it is washed out with plenty of tap water until the washings no longer redden blue litmus paper. It is next fixed to a Liebig's condenser by means of a piece of stout india-rubber tubing, that has been previously boiled for some time in a $\frac{E}{2}$ solution of sodic hydrate, in order to free it from any traces of ammonia. About half a dozen pieces of recently-ignited pumice stone each about the size of a pea are now introduced into the retort, which serve to prevent "bumping," and 500 cc. of water free from ammonia poured in. Any water present in the condenser is allowed to run off, so as to allow *steam* to pass through to clear out all traces of ammonia. A Bunsen burner is lit under the retort and the water made to boil; steam will soon find its way through the condenser, and after it has been steaming for about half an hour, the cold water is turned on to the condenser and a Nessler cylinder (these are colourless glass cylinders usually 6 ins. high and 1½ ins. in diameter, and marked at 50 cc.) is now put under the delivery tube. When 50 cc. have come over, add to it 2 cc. of the Nessler solution and mix by pouring backwards and forwards into an empty Nessler glass. On placing on a sheet of white paper there should be no yellow coloration produced; if there should be, continue the distillation with pure water until the apparatus is free from traces of ammonia.

If the sample of water under examination contain much in the way of suspended matter, it will be wise to filter it before subjecting it to the ammonia process, through a filter that has been previously washed with the pure distilled water till free from ammonia. Any residual water having been syphoned out of the retort, 500 cc. of the clear sample are introduced, and a clean empty Nessler glass placed under the delivery tube of the condenser. Distillation is now proceeded with, and when 50 cc. of distillate have been collected in the Nessler glass, it is removed and an empty one put in its place. It now becomes necessary to ascertain how much free ammonia is present in the first 50 cc. of distillate; for this purpose run in 2 cc. of the Nessler solution from a pipette, well mix and place on a sheet of white paper. Observe the intensity of colour produced. If it is of a faint yellow tint there is not much in the way of "free" ammonia present, but if it appears of a rich reddish brown colour a fair quantity is present, and sometimes when waters are highly contaminated with sewage, there is so much ammonia present as to occasion a brownish precipitate. In all such cases it is better to take a fraction of the 500 cc. for distillation, diluting to 500 cc. with the pure distilled water, previous to distillation. A burette graduated in 0·1 of a cc. is filled with the dilute standard ammonia solution, and a definite volume of it, say 3 cc., run into a clean Nessler glass; this is diluted with the pure distilled water to 50 cc., 2 cc. of Nessler solution added and well stirred; it is now placed beside the 50 cc. of distillate, and the colours compared by looking down through the liquids. If the solutions are not of equal depth of colour a fresh standard must be made, using more or less, as the case may demand, of the dilute ammonia solution, and Nesslerizing as before until a certain quantity used produces exactly the same colour as the sample. The "free" ammonia is estimated in the first 50 cc. of distillate only, $\frac{1}{3}$ of the quantity thus found being added on; this being the quantity that is invariably present in the next 150 cc. that are distilled over, before the introduction of the potash and permanganate solution for the determination of the albuminoid ammonia. Supposing that 2·5 cc. of the dilute standard ammonia solution were required for the first 50 cc. of distillate, that is, $2{\cdot}5 \times {\cdot}01 = {\cdot}025$ milligram of ammonia, then the amount of free ammonia present in 500 cc. of sample will be $\frac{{\cdot}025}{3} + {\cdot}025 = {\cdot}0333$ milligram, or ${\cdot}0333 \times 2 = {\cdot}0666$ parts per million, or ${\cdot}0666 \times {\cdot}07 = {\cdot}00466$ grain per gallon.

The Albuminoid Ammonia.—After the 200 cc. have been distilled over and the free ammonia estimated as described, 50 cc. of the potash and permanganate solution are introduced into the retort, and the distillation proceeded with as before, Nesslerizing each 50 cc. that comes over, three of which are usually sufficient.

The following is an example of the determination of the free and albuminoid ammonia in a sample of water contaminated with a little sewage:—

FREE AMMONIA.

500 cc taken.

1 cc. of standard dilute AmCl = ·01 milligram of NH_3.
1st 50 cc. distillation required 6·3 cc. = ·063 milligram NH_3.
Correction $\frac{1}{3}$ = ·021 " "
Total . ·084

·034 × 2 = 0·168 part per million,

or

·168 × ·07 = ·01176 grain per gallon.

ALBUMINOID AMMONIA.

1st 50 cc. required 5·0 cc. = ·050 milligram NH_3.
2nd " " 1·8 cc. = ·018 " "
3rd " " 0·8 cc. = ·008 " "
Total ·076 " "
Subtracting NH_3 found in potash solution ·005 " "
·071 " "

·071 × 2 = 0·142 part per million,

or,

0·142 × ·07 = ·00994 grain per gallon.

Remarks.—When the free ammonia in waters exceeds ·08 part per million, it is often due to the fermentation of urea, into carbonate of ammonia, owing to the presence of urine, &c., in the water—the chlorine being likewise high; but there are waters which give a comparatively high proportion of free ammonia, with little albuminoid ammonia, and low chlorine. In such cases the free ammonia may be safely considered of saline or mineral origin rather than from a decomposition of organic pollution. When the albuminoid ammonia is high, and the free ammonia and chlorine low, it is diagnostic of the presence of organic matter of vegetable origin. In such cases the odour given off when the total solid residue is gently ignited, will be of a peaty or some such characteristic vegetable odour. With regard to the amount of "free" and "albuminoid" ammonia present, that admits of a water being passed or condemned for drinking purposes, Professor Wanklyn states—

"If a water yield 0·00 part of albuminoid ammonia per million, it may be passed as organically pure despite of much free ammonia and chlorides; and if indeed the albuminoid ammonia amount to ·02 or to less than 0·05 parts per million, the water belongs to the class of very pure water. When the albuminoid ammonia amounts to ·05, then the proportion of free ammonia becomes an element in the calculation, and I should be inclined to regard with some suspicion a water yielding a considerable quantity of free ammonia along with more than 0·05 part of albuminoid ammonia per million. Free ammonia, however, being absent, or very small, a water should not be condemned unless the albuminoid ammonia reaches something like 0·10 part per million. Albuminoid ammonia above 0·10 per million begins to be a very suspicious sign; and over 0·15 ought to condemn a water absolutely. The absence of chlorine or the absence of more than 1 grain of chlorine per gallon, is a sign that the organic impurity is of vegetable rather than of animal origin; but it would be a great mistake to allow water highly contaminated with vegetable matter to be taken for domestic use."

The Nitrates.—The nitrogen present as nitrates and nitrites is to be determined as directed on page 206. Nitrates may be present in waters owing to the solvent action of the water on the nitrates present in the geological strata through which the water passes, or to the decomposition of nitrogenous organic matter present, with the production of nitric and nitrous acid, which in turn act upon the carbonates with the formation of nitrates and nitrites. Dr. Frankland lays much importance on the presence of nitrates in waters as indicating past pollution, and although the organic matter is thus destroyed, it may still contain living animalculæ capable of propagating disease; on the other hand, Dr. Wanklyn states that "presence or abundance of nitrates does not show defilement by means of sewage, and deficiency of nitrates does not show absence of defilement." Whether or not the presence of nitrates is a measure of past or present organic pollution of a water is immaterial, since we have other means of determining its *actual* presence; and, moreover, knowing the filth that enters such rivers as the Thames every day previous to its distribution by the water companies for consumption, and that such waters are pronounced as being wholesome, it seems to be of secondary consideration what the condition of a water *was* so long as it comes up to the ideal of organic purity as it *is*.

The Nitrites.—A qualitative test for the presence of nitrites may be of interest in ascertaining whether any nitrogenous organic matter present is still decomposing, &c. The presence of nitrites may be quickly determined by the following method: 100 cc. of the water are taken, acidulated with 1 cc. of 5 E H_2SO_4, 5 cc. of starch and potassic iodide solution (1 grm. of starch and 0·2 grm. of iodide in 100 cc.) added, and well stirred. If any considerable quantity of nitrites be present, a blue colour of iodide of starch will be at once produced, but if present only in minute quantity, the colour will not be produced until after standing for some time.

The Hardness.—The hardness may be determined as directed on page 200. It is always desirable to obtain *soft* water for laundry and toilet use, since it is more efficient and economic to use as a detergent than hard water, which would require artificial means for softening, such as boiling, or the use of excess of soap or soda. Where the supply of water is excessively hard, rain water, if it can be procured, would be far superior for such purposes. As a general rule, the hardness of a water increases with the total solids. It is said that persons who are predisposed to such diseases as *calculus* and *bronchocele* are liable to propagate it if they habitually drink very hard water.

Poisonous Metals.—A drinking water should always be tested for lead. The presence of this metal is due to the solvent action of soft or faintly acid waters upon lead piping, which is often used to convey the water; and a test should likewise be made for copper and iron. A useful qualitative test for copper, lead, and iron collectively is as follows:—Pour 100 cc. of the water into a porcelain basin, add a drop of 5 E ammonium sulphide, and stir. If any copper, lead, and iron be present, a more or less dark colour will be produced, owing to the formation of the sulphides of the metals. If the colour produced is due to iron only, a drop of 5 E HCl will remove it, but will unaffect any sulphide of lead or copper present.

If lead is detected by this means it should be determined by Dr. Miller's colorimetric method, as follows:—A standard solution of crystallized lead acetate is first prepared by dissolving 0·1831 grm. of the salt in a litre of distilled water, each cubic centimetre of such solution containing ·0001 grm. of metallic lead; 70 cc. of the sample are poured into a white porcelain dish, two drops of 17 E acetic acid, and 5 cc. of a saturated solution of sulphuretted solution added, and stirred.

It now becomes necessary to imitate the test thus produced by a known volume of the standard lead solution; for this purpose pour about 65 cc. of distilled water into a similar porcelain dish, add 5 cc. of sulphuretted hydrogen water, stir, fill a burette with the standard lead solution, and allow it to run gradually into the distilled water, until the tests in both dishes are alike. Each cubic centimetre of lead solution required will represent $\frac{1}{10}$ of a grain of lead present in a gallon of water. Owing to the poisonous nature of water containing small quantities of lead, all waters containing $\frac{1}{10}$ of a grain or upwards of the metal per gallon should be rejected for drinking purposes.

Copper.—This metal may be estimated in a manner similar to the above, the solution for comparison being prepared by dissolving 0·3929 grm. of crystallized sulphate of copper in a litre of water, each cc. containing ·0001 grm. Cu.

Arsenic, Barium, Zinc, and Manganese, &c. sometimes find their way into waters, especially in those mining and chemical works districts where the metals are obtained and worked.

The Colour of Waters.—Good drinking water should be free from colour. The colour of a sample of water can be best judged, by filling a colourless glass cylinder about two feet high with the sample and looking down through the liquid on to a sheet of white paper, making a comparison with distilled water contained in a similar cylinder.

The Suspended Matter.—The suspended matter if present in appreciable quantity, may be estimated as described on page 198, and it would be interesting to make, if possible, a microscopical search for any low forms of animal or vegetable life that it may contain.

Classification of Drinking Waters.—Dr. Frankland makes the following classification of waters for domestic purposes:—

Wholesome	1. Spring water	Very palatable.
	2. Deep well water	
	3. Upland surface water	Moderately palatable.
Suspicious	4. Stored rain water	
	5. Surface water from cultivated land	Palatable.
Dangerous	6. River water to which sewage gains access	
	7. Shallow well water	

The following is a classification of waters according to their softness:—

1. Rain water.
2. Upland surface water.
3. Surface water from cultivated land.
4. Polluted river water.
5. Spring water.
6. Deep well water.
7. Shallow well water.

PART V.

OILS.

In this section it is proposed to deal with oils in three divisions—(1) Oils for Lubricating, (2) Illuminating, (3) those adapted for special purposes. There is, of course, no hard and fast rule for the legitimate classification of oils in this manner, since some oils, such as rape, would come under the three divisions; but it will be found convenient.

(1) Lubricating Oils.

A lubricating oil has for its object the reduction of friction and the prevention of an undue development of heat. The result of friction is to abrade solids, heat being given out both in solids and liquids; the amount being the exact measure of the power wasted as a consequence of the friction. The amount of heat generated for each 772 foot pounds of work thus expended, is equivalent to the amount of heat necessary to raise the temperature of 1 lb. of water 1° F. Lubricants may be solid, semi-solid, or liquid, and the "body" or "viscosity" of them should be proportional to the pressure which will be exerted upon them. They may be fatty (animal or vegetable), mineral, or compound. Soap is a constituent of railway grease, and graphite and steatite are sometimes used for heavy machinery.

The following list shows the best purpose to which the various lubricants can be applied:—

For steam cylinders . .	Heavy mineral oils, lard, tallow, rape oil, &c.
Ordinary machinery . .	Rape oil, lard oil, tallow oil, and medium mineral oils.
For very great pressures with slow speed . . .	Graphite, soapstone, &c.
For great pressures with slow speed . . .	Tallow, lard, palm oil, grease, &c.
For heavy pressures and high speeds . . .	Sperm oil, rape oil, castor oil, medium mineral oils.
For light pressures and high speed.	Sperm, refined petroleums, cottonseed, rape, olive, and mineral oils.
Watches, clocks, &c. .	Light mineral oils, clarified sperm, neatsfoot, olive and porpoise.

It may be stated, however, that in all cases where possible, a mineral oil of suitable "body" should be used, especially for cylinders, the high temperature of which decomposes fatty oils, with the production of free fatty acids, which attack copper, iron, &c., forming metallic soaps, which cause much damage to fittings, &c.

In judging of the suitability of a lubricating oil, the purpose for which it is proposed to be used being first ascertained, the following points must be determined:—

(1) The "body" or viscosity of the sample at the temperature at which it is proposed to be used.

(2) The specific gravity at 15° C., or in some cases at the temperature of boiling water.

(3) The "flashing point," or the temperature at which the vapour evolved becomes ignited on the application of a flame.

(4) The percentage of free, fatty, or mineral acids present.

(5) The percentage of fatty and mineral oils.

(6) The loss the oil suffers when exposed for about twelve hours to the temperature at which it is proposed to work.

(7) The amount, if any, of mineral and organic suspended matters present.

(8) The temperature at which the oil solidifies or congeals.

(9) A test to ascertain the liability of the oil to gum.

(10) The extent of the corroding action of the oil on pieces of steel, brass, bronze, &c.

Determination of Viscosity.—The viscosity or body of an oil, although not an exact measure of unctuousness or greasiness, is one of the most important factors in judging of its suitability for any special class of lubrication. The viscosity should be proportional to the pressure exerted upon the oil—that is to say, an oil for any specific purpose should not be so thick as to cause undue fluid friction, nor so thin that it is squeezed out, leaving the bearings, &c., dry.

The viscosity of an oil decreases considerably with an increase in temperature; mineral oils in this respect decreasing, through a given range of temperature, far more than the majority of fatty oils. There is no fixed ratio between the viscosities of oils at the ordinary temperature, and the viscosities at an elevated temperature, *i.e.*, two different oils having identical viscosities at 15° C. would in all probability have different viscosities at a temperature, say, of 100° C. The specific gravity of an oil, although valuable as a means in some cases of identification, is no criterion of the viscosity. The best form of apparatus suitable for obtaining viscosities at different temperatures, is that devised by Mr. Boverton Redwood, F.R.S.E., &c. Each apparatus is standardized by Mr. Redwood himself, being so adjusted that 50 cc. of a standard average rape oil take 535 seconds to flow out at a temperature of 15·5° C., distilled water taking 25·5 seconds to flow out under the same conditions. The following is a sketch (Figs. 51 and 52) of the apparatus* and *modus operandi* of the process :—

The viscosity tube A is of electro-plated copper 3½ inches

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* The standardized apparatus can be obtained from Townson & Mercer, 89, Bishopsgate Street, E.C., and James How and Co., 73, Farringdon Street, E.C.

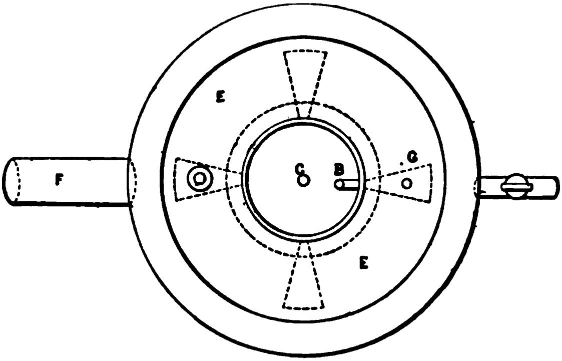

Fig. 51.—Horizontal Section.

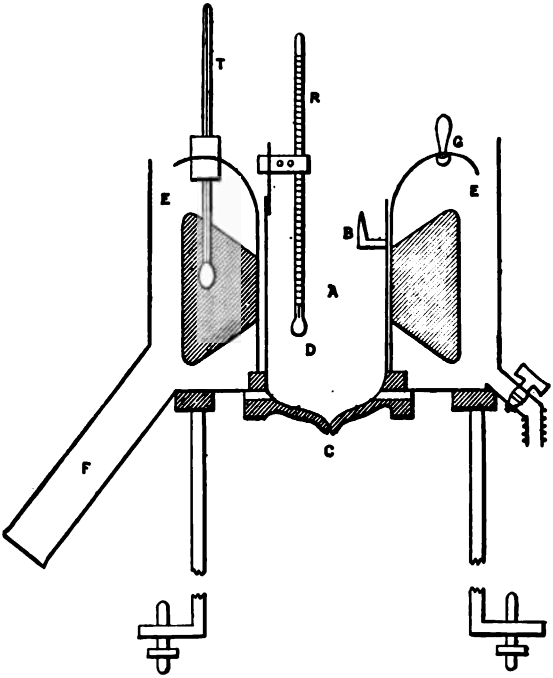

Fig. 52.—Vertical Section.

Redwood's Apparatus for Determining Viscosity of Oil.

high and 1⅞-inch internal diameter. The orifice C is of agate, of definite bore and limited protuberance, and a plug of electro-plated copper suspended to a wire is supplied with the apparatus, in order to close it while the oil is being run in and adjusted. The outer jacket E is made of copper, in which water, oil, or any other liquid can be maintained at a definite temperature by the application of a flame to F. The temperature is made uniform by means of the paddles which can be revolved with the handle G.

In order to obtain viscosities at elevated temperatures, E is filled with mineral oil of high-boiling point, and heated with a Bunsen until the desired temperature is attained, which is kept constant by the regulation of the flame, and the oil mixed by means of the paddles. The sample of oil to be tested is heated in an air bath to the required temperature, and poured into the viscosity tube A until the surface of the oil just reaches the point of the gauge B. When it is assured that the temperatures registered by the thermometers T and R are the same, a small narrow-necked flask holding 50 cc. to a mark made on its neck, is placed under the orifice in a vessel containing a liquid heated to the same temperature as the oils. The plug is then taken out of C, and the number of seconds that 50 cc. of the oil take to run out, is noted.

The temperature at which the viscosity of an oil should be determined, depends upon the temperature at which it is proposed to be used. With oils proposed to be used for ordinary machinery, the viscosities should be determined at temperatures of 15°, 30°, and 60° C.; while for cylinders, at temperatures of 100°, 120°, and 150° C.

The following interesting table by Redwood,* gives the comparative rates of flow of various oils for every rise of 10° F., by the above standard apparatus:—

---

* *Vide* "Journal Soc. Chem. Ind.," v. 128.

Temperature °F.	Refined Rape Oil.	Refined Rape Oil.	Refined Rape Oil.	Refined Rape Oil.	Beef Tallow.	Sperm Oil.	Neatsfoot Oil.	American Mineral Oil. Sp. gr. ·885.	American Mineral Oil. Sp. gr. ·913.	American Mineral Oil. Sp. gr. ·923.	Russian Mineral Oil. Sp. gr. ·909.	Russian Mineral Oil. Sp. gr. ·915.	Russian Mineral Oil. Semi-solid.
50	712·5	—	—	—	—	—	620	145	425	1030	2040	2520	—
60	540	—	—	—	—	177	470	105	295·5	680	1235	1980	—
70	405	406	405·5	407	—	137	366	90	225	485	820	1320	—
80	326	—	—	—	—	113	280	73	171	375	580	900	—
90	260	—	—	—	—	96	219	63·5	136	262	426	640	—
100	213·5	—	—	—	—	80·5	175	54	111	200	315	440	1015
110	169	—	—	—	—	70·5	147·5	50	89·5	153	226	335	739·5
120	147	146	147	147·5	—	60·5	126	47	78	126	174	245	531
130	123·5	—	—	—	—	57	112	44·75	63·5	101	135·5	185	398·5
140	105·5	106·5	106·5	106	—	51	88·5	41	58	82	116	145	317·5
150	95·5	—	—	—	—	49	75·5	37·5	52	70·5	95	115	250
160	85	—	—	—	—	47·5	70	—	46	63·5	83·5	93·5	200
170	76	—	—	—	—	46	62	—	—	58	70·5	77·5	161
180	69	—	—	—	—	44·5	56·5	—	—	52·5	61·5	67·5	134·5
190	64·5	—	—	—	—	43	53	—	—	47	56·5	61	115·5
200	58·5	57·5	57·5	58·5	54·5	42	50·5	—	—	42	48	54	99
210	54	—	—	—	—	41	48·5	—	—	40	—	—	85
220	54	—	—	—	—	39	47	—	—	38	—	—	77
230	47·5	—	—	—	—	37	46	—	—	—	—	—	70·5
240	45·5	—	—	—	—	36	44·5	—	—	—	—	—	64·5
250	43·5	—	—	—	40	35	44	—	—	—	—	—	59
260	Viscosity slightly above the average.	—	—	—	—	34	43·5	—	—	—	—	—	54
270		—	—	—	—	33	43	—	—	—	—	—	48·5
280		—	—	—	—	32	41·5	—	—	—	—	—	46·5
290		—	—	—	—	31	41	—	—	—	—	—	46
300		—	—	—	—	30	38	—	—	—	—	—	42·5

The following table gives the number of seconds that 50 cc. of oil (mineral) take to run out of Redwood's Viscosimeter at various temperatures, and the purposes for which such oils the writer has found to be most suitable :—

	At 60° F.	140° F.	180° F.	220° F.
	Secs.	Secs.	Secs.	Secs.
Cylinders, valves, and heavy machinery	—	—	95	65
Medium machinery, carriage oils, boxes, &c.	540	105	70	54
Light machinery, spindles, &c. .	290	52	30	15

The Fluidimeter.—This instrument was designed by the Author for ascertaining the fluidity or viscosity of several oils at the same time. It consists (Fig. 53) of a copper bath, 14 in. long, 9 in. wide, and 1½ in. deep, provided with a stout silver-plated gun-metal plate containing six cups connected with graduated grooves, and swung upon elevated bearings from two studs protruding from the centre of the longitudinal sides, and fixed by means of thumb screws. A protractor is fixed on the side of the bath, so that the latter can be tilted at any desired angle. At the top of the bath are two holes, one for a thermometer and the other for pouring in oil or water, to be maintained at any desired temperature by a bunzen burner; a stout glass plate keeps away air currents.

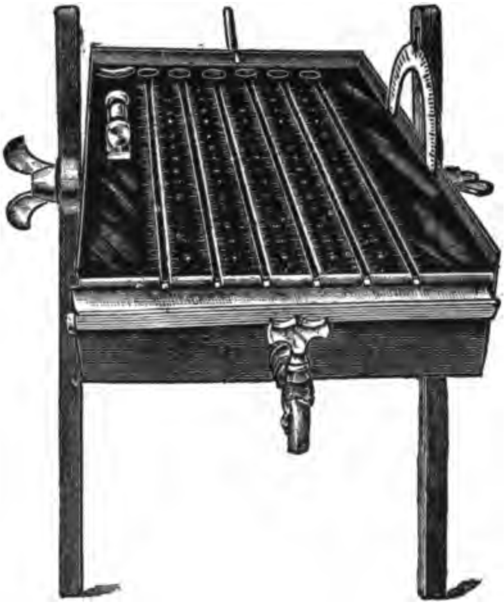

FIG. 53.—PHILLIPS'S FLUIDIMETER.

The rate of flow of various oils on this machine at various temperatures has been found to be practically identical with those obtained by Redwood's Viscosimeter.

Gibbs' Viscosimeter.—The writer is indebted to Dr. Stillman, of the Hoboken Technological Institute, for a communication on a new form of viscosimeter used on the Chicago, Milwaukie and St. Paul Railroad, designed by Mr. Gibbs,

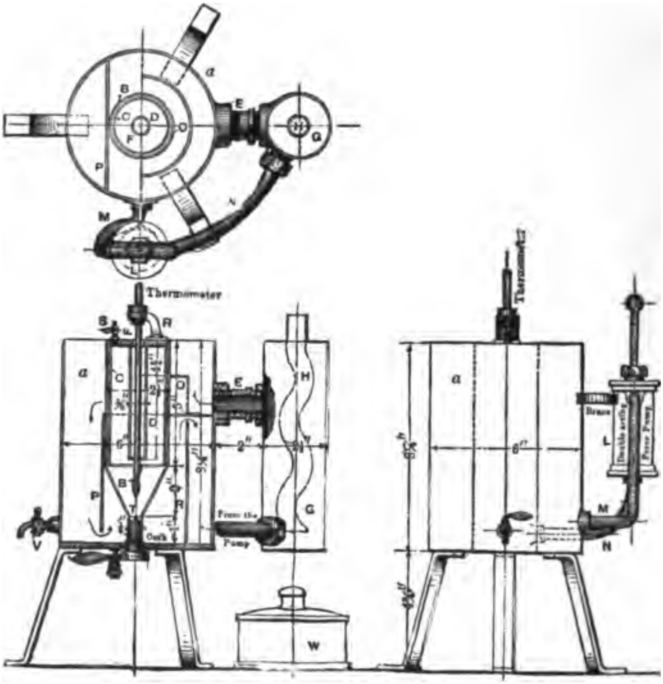

FIG. 54.

to overcome some of the objectionable points in existing forms of viscosimeters. The advantages of its use being :—

(1.) To have a large body of hot oil as a bath surrounding the oil to be tested in order to keep the latter at a perfectly even temperature.

(2.) To apply a forced circulation to the bath by means of a double action pump, to insure equality of heat in all parts.

(3.) To deliver the oil to be tested at the orifice under a constant head, which is accomplished by means of a pneumatic trough.

(4.) To supply convenient means for accurately measuring the temperature of the oil.

The preceding page contains a sketch, Fig. 54, of the apparatus.

The large reservoir, *a*, is of copper, with heavy brazed bottom. This contains the cylindrical inside chamber with conical bottom B. At the lower end of this is the gauged aperture T. Inside of this chamber fits the inverted reservoir C, holding the oil to be tested. In the interior of this chamber is a tube, D, extending nearly to the bottom of the same. This tube admits air to determine the head of the oil, and also to admit the thermometer F. The outside bath, *a*, contains the deflector plates, O, P, and R, to obtain proper mixing of the bath. The heating of the bath is done by a lamp, W, set underneath the separate heating chamber G. The size of the orifice at T is $\frac{1}{16}$ inch.

Specific Gravity.—The specific gravity of oils is usually determined at a temperature of 15° C., and in the case of solid fats, at any convenient temperature above their melting points. A quick and fairly accurate specific gravity of oils fluid at the ordinary temperature, may be obtained by the hydrometer (Fig. 55). The oil having been brought to the required temperature in a glass cylinder, the hydrometer is dropped into it; when it stops sinking, the point of the graduated stem, which the tip of the meniscus of the oil just touches, is taken as the specific gravity. In the examination of oils, it will be convenient to keep three hydrometers, graduated from ·700 to ·800, ·800 to ·900, and ·900 to 1·000 respectively. Some oils are too thick to allow their specific gravities being taken with the hydrometer at the ordinary temperature; in such cases the specific gravity bottle (Fig. 56) may be used—one holding about 50 cc., with a fine hole drilled through the stopper, is convenient. The weight of the empty dry bottle being first ascertained, it is filled with distilled water at 15° C., and again weighed; and by subtracting the weight of the bottle, the weight of the water it holds is thus obtained. The water having been poured away and the bottle dried, the sample of oil at a temperature of 15° C. is poured in, and the bottle stoppered, cleaned, dried and weighed. The weight of the oil thus obtained (by subtracting the weight of the bottle from that of the bottle plus oil), divided by the weight of water the bottle holds, will give the specific gravity of the oil.

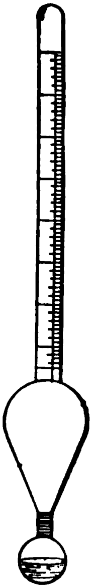

FIG. 55.

In determining the specific gravity of the solid fats at elevated temperatures, Sprengel's specific gravity tube (Fig. 57) is useful. The weight of water at 15° C. it holds having been first ascertained, the melted fat is sucked into it and placed into a beaker of water at the desired temperature; after being immersed sufficiently long for the oil to acquire the temperature of the water, the caps are put on each of the ends of the capillary tubes, dried, cooled and weighed, from which the specific gravity is calculated; water at 15° C. being taken as unity. It sometimes happens that in extracting solid fats, &c., from substances, there is insufficient to make a determination of the specific gravity by the usual methods; in such cases it can be obtained fairly accurately by floating a thin piece of the fat on distilled water at the desired temperature, and adding alcohol, until on moving the fat about with a glass rod, it will stop in any desired part of the liquid. The specific gravity of the liquid being ascertained with the hydrometer or bottle, the specific gravity of the fat is thus obtained.

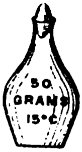

FIG. 56.

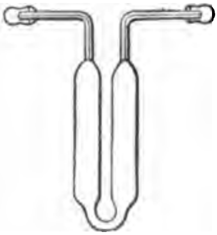

FIG. 57.

The following is a table of specific gravities, at 15° C., by Stillwell, of various kinds of animal and vegetable oils :—

Sperm, bleached winter	·8813
Sperm, natural winter	·8815
Elaine	·9011
Red, saponified	·9016
Palm	·9046
Tallow	·9137
Neatsfoot	·9142
Rape-seed, white winter	·9144
Olive, light greenish yellow	·9144
Olive, dark green	·9145
Pea-nut	·9154
Olive, virgin, very light yellow	·9163
Rape-seed, dark yellow	·9168
Olive, virgin, dark clear yellow	·9169
Lard, winter	·9175
Sea elephant	·9199
Tanner's (Cod)	·9205
Cotton-seed, raw	·9224
Cotton-seed, refined yellow	·9230
Salad (cotton-seed)	·9231
Labrador (Cod)	·9237
Poppy	·9245
Seal (natural)	·9246
Cocoa nut	·9250
Whale, natural winter	·9254
Whale, bleached winter	·9258
Cod liver, pure	·9270
Seal, racked	·9286
Cotton-seed, white winter	·9288
Straits (Cod)	·9290
Menhaden (dark)	·9292
Linseed (raw)	·9299
Bank (cod)	·9320
Menhaden (light)	·9325
Porgy	·9332
Linseed, boiled	·9411
Castor, pure, cold pressed	·9667
Resin, third run	·9887

The mineral lubricating oils have specific gravities ranging from ·850 to ·925.

The following is a table of specific gravities and yield of various products on the distillation of 100 gallons of Russian petroleum :—

	Specific Gravity.	Gallons.
"Benzine"	·725	1
"Gasolene"	·775	3
Kerosene	·822	27
Pyronaphtha	·858	12
Lubricating oil	·890-·905	27
Cylinder oil	·915	5
Vaseline (not a direct product)	·925	1
Residuum and loss	—	24
		100

The Flashing Point.—The flashing point, or the temperature at which an oil gives off inflammable vapour, can be determined by the Abel flash-point apparatus, described on page 261 ; but the cup must be taken out of the water bath and placed in a copper air bath, since the flash point of most lubricating oils is above the temperature of boiling water. A small flame is kept under the apparatus, and the oil tested at every rise of one degree, as in the case of the petroleums. The flash point of a lubricating oil must be higher than the temperature at which it is proposed to work. For cylinders, &c., oils should not flash below 200° C., and for ordinary machinery not below 149° C. The light Scotch shale oils flash at from 130° to 185° C., while the darker variety used for heavy machinery, cylinders, &c., flash at from 180° to 230° C.

Determination of Free Fatty and Mineral Acids.—Free fatty acids present in fatty oils are brought about by the decomposition of the glycerides, which thus become rancid. The free fatty acids, present in oils intended for lubricating, should not exceed 5 per cent., owing to their corroding action on bearings, &c. ; while for burning purposes, 3 per cent. should be the maximum quantity present, otherwise the wicks become charred. Free mineral acids are sometimes present in very small quantities, owing to their imperfect elimination during refining; they have a very corroding action on bearings, &c. The free fatty acids may be determined as follows: Weigh out 50 grms. of the sample (or in the case of palm oil, 5 grms.) into a 200 cc. flask, and pour on to it 100 cc. of neutral methylated alcohol, to which three drops of an alcoholic solution of phenolphthalein (1 in 30), and one drop of E NaHO has been previously added; the pink solution thus produced soon becomes colourless on agitating with the oil if any free fatty acid be present. After agitating for some time to ensure the complete solution of the free acids, a *standard* E solution of sodic hydrate is gradually run in from a burette, stirring, until the last drop added produces a permanent pink coloration on agitating for five minutes. Note the volume of standard soda solution thus required. Each cc. of solution required, corresponds to 0·282 grm. of free oleic acid ($C_{18}H_{34}O_2$), 0·256 grm. free palmitic acid ($C_{16}H_{32}O_2$), or 0·284 grm. of free stearic acid ($C_{18}H_{36}O_2$), as the case may be.

Free mineral acids may be detected by agitating 50 cc. of the oil with about 30 cc. of distilled water free from acid; and on adding an alcoholic solution of methyl-orange to the aqueous extract, a more or less pink coloration will be produced if any free mineral acid be present. An idea of the amount could be ascertained by titrating with standard $\frac{E}{10}$ NaHO. The nature of the free mineral acids could be determined by dividing an aqueous extract of 50 cc. of the sample into three equal parts, adding $BaCl_2$ to one for H_2SO_4, $AgNO_3$ to another for HCl, and sulphate of brucine, and 36 E H_2SO_4 to another for HNO_3. When free mineral acid is present in oils it is mostly sulphuric acid. A very delicate test for the detection of minute quantities of free sulphuric acid is to evaporate an aqueous extract of about 20 grms. of the oil, to dryness, with an addition of a few crystals of white cane sugar, and on heating the residue for some time in a water bath, a more or less browning or blackening will be produced.

The following table by Watson shows the corroding action of various oils upon iron and copper;—

Oils.	Iron dissolved in 24 days.	Copper dissolved in 10 days.
Almond	·0040 grain	·1030
Castor	·0048 ,,	—
Colza	·0800 ,,	·0170
Lard	·0250 ,,	—
Linseed	·0050 ,,	·3000
Neatsfoot	·0875 ,,	·1100
Olive	·0062 ,,	·2200
Paraffin	·0045 ,,	·0015
Seal	·0050 ,,	·0485
Sperm	·0460 ,,	·0030

The extent of the action of oils on metals, is directly proportional to the amount of free fatty acids present, and most of the fatty oils act more rapidly upon copper than on iron.

The following table * shows the minimum, maximum, and mean percentage proportions of free fatty acids in various oils :—

A. Table oils obtained by pressing.
B. Expressed commercial oils.
C. Oils extracted by light petroleum.

	A.			B.			C.		
	min.	max.	mean	min.	max.	mean.	min.	max.	mean.
Rape-seed oil . .	0·53	1·82	1·19	0·52	6·26	2·88	0·77	1·10	0·93
Olive oil . . .	—	—	1·66	3·87	27·16	12·97	—	—	—
Poppy oil . . .	0·70	2·86	1·92	12·87	17·73	15·37	2·15	9·43	4·72
Earth-nut oil . .	0·85	3·91	1·94	3·58	10·61	6·52	0·95	8·85	4·02
Oil of sesame . .	0·47	5·75	1·97	7·17	33·13	17·94	2·62	9·71	4·89
Cotton-seed oil . .	—	—	0·15	0·42	0·50	0·46	—	—	—
Oil of mustard . .	—	—	—	0·68	1·02	0·85	—	—	—
Castor oil . . .	—	—	—	0·62	18·61	9·28	1·18	5·52	2·78
Linseed oil . .	—	—	—	0·41	4·19	1·57	—	—	—
Candle-nut oil .	—	—	—	—	—	56·45	—	—	—
Palm kernel oil .	—	—	—	3·3	17·65	6·91	4·17	11·42	8·49
Palm oil (old) .	—	—	—	—	—	50·82	—	—	—
Cocoanut oil . .	—	—	—	3·03	14·35	7·92	1·00	6·31	4·26
Illipe fat (3 years old)	—	—	—	—	—	28·54	—	—	—
Niam fat . . .	—	—	—	—	—	—	14·40	34·72	24·56
Bicuhyba fat . .	—	—	—	—	—	18·55	—	—	—
Japan wax . . .	—	—	—	—	—	9·25	—	—	—

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* Noerdlinger, "Zeit. Anal. Chem.," 28, 183-187.

Determination of Fatty and Mineral Oils.—Mixtures of fatty and mineral oils are now much used in lubrication, and it becomes frequently necessary to determine the proportion of each present. In order to effect their separation, advantage is taken of the fact that, while fatty oils are decomposed and converted into soaps by the action of a solution of potash or soda, the mineral oils do not suffer any apparent change by such treatment. The following is the *modus operandi* of the process :—

Weigh out 5 grms. of the sample into a 5-oz. wide-mouth stout glass bottle, provided with an indiarubber stopper. Pour into it 26 cc. of 1½ E alcoholic potassic hydrate solution, and place into a bath of cold water. A Bunsen is lit under the bath, and when the alcoholic solution is seen to boil, the bottle is taken out, and the stopper fixed and tied down with wire; it is now again placed into the bath, the water made to boil, and the bottle occasionally shaken; after heating thus for about half an hour, the bottle is taken out, allowed to cool, the contents poured into a porcelain basin, and evaporated nearly to dryness over a water bath; 60 cc. of warm water are now poured into the bottle and well shaken in small quantities at a time, so as to remove any adhering matter, poured into the dish, and stirred with a glass rod until the soap goes into solution: any mineral oil present will float, or remain in an emulsive condition with the solution of soap. The whole is now carefully poured into a globular or cylindrical separator (Figs. 58 and 59), and the dish and bottle washed out with water and about 50 cc. of methylated ether (using small quantities at a time), into the separator; the stopper is now put in, the contents violently shaken up for about ten minutes, and the mixture allowed to separate; sometimes there is a little difficulty in the separation, an emulsion being formed. In such cases an addition of 2

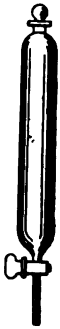
FIG. 58.

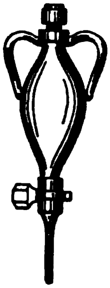
FIG. 59.

or 3 cc. of absolute alcohol with a rotatory motion puts matters all right. When complete separation is ensured, the aqueous solution is tapped off into a second separator, the aqueous solution shaken up with 20 cc. more ether, allowed to separate, the aqueous solution tapped off as before, and the ethereal extract added to the main portion, which in turn is washed three times with 20 cc. of water, and the washings added to the main aqueous solution. The ethereal solution of the mineral oil is now tapped off into a weighed flask attached to a Liebig's condenser, and the ether distilled off; the residual mineral oil is then dried in a water oven, air being pumped into it occasionally to get rid of the last traces of water, which was taken up by the ether; after which it is cooled and weighed, and dried again until no further loss occurs. If the mineral oil present be light, and of low boiling point, considerable loss would be incurred by its evaporation by this process; in such cases it would be best to determine by difference, or dry at a lower temperature where practicable. The specific gravity, congealing, and melting points, would convey an idea of the nature and source of the mineral oil present. The aqueous solution contained in a separator, which now contains the oil in the form of soap, is acidulated with 10 cc. of 10 E HCl, the fatty acids being thus precipitated. 40 cc. of methylated ether are added, and the mixture well shaken up; the fatty acids are thus dissolved by the ether. After complete separation occurs, the acid solution is tapped off, and the ethereal solution washed three times with about 20 cc. of water, after which it is run off into a weighed flask, the separator being washed out with a little ether. The flask is attached to a Liebig's condenser, the ether distilled off, and the residual fatty acids dried in the water oven until no further loss of weight occurs. On adding 5 per cent. for loss of glycerine, to the weight of the fatty acids thus obtained, this gives practically the proportion of fatty oil present. In order to identify the oil, obtain its specific gravity, melting point, odour on ignition, and if resin is indicated estimate by Gladding's process, given on page 295.

Rapid Method for Separating Fatty Oils from Mineral Oils.—Gripper* has found that an extension of Kættstorfer's process for the determination of the saponification equivalent can be applied to a quick determination of fatty and mineral oils in a compound oil. 2½ grams of the sample are saponified, as described on page 251, with 25 cc. of standard $\frac{E}{2}$ alcoholic potash solution; when saponification is complete, the excess of KHO is titrated with standard $\frac{E}{2}$ HCl, using phenolphthalein as indicator. A blank experiment is made to ascertain the strength of the alcoholic potash used, after which the percentage of potash absorbed is readily calculated. The following are the KHO absorption figures for oils generally used in mixtures for lubricating purposes:—

	KHO absorption.	Saponification equivalent.
Cotton seed . .	22·27 per cent.	252
Rape . . .	20·03 „	280
Neatsfoot . . .	18·70 „	300
Lard	19·30 „	290
Tallow . . .		
Castor . . .	17·90 „	313

The mean percentage of KHO absorbed by these oils is 20·08 per cent., then $\frac{100}{20·08} = 4·98$ parts oil saponified by 1 part of KHO; therefore if the number of cc.'s of standard $\frac{E}{2}$ HCl (corresponding to KHO neutralized) be multiplied by 5·587 (= ·02805 × 40 × 4·98) the approximate percentage of fatty oil is obtained.

The following table by Hübl gives the melting and congealing points of fatty acids from various oils and fats, and will serve as a good guide as to the nature of the oil present:—

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* *Chem. News*, vol. lxv., No. 1677.

Fatty Acids from—	Melting point. °C.	Congealing point. °C.
Olive oil	26	21·2
Almond oil	14	5·0
Arachis oil	27·7	23·8
Rape oil	20·1	12·2
Cottonseed oil	37·7	30·5
Sesamé oil	26·0	22·3
Linseed oil	17·0	13·3
Poppy oil	20·5	16·5
Hempseed oil	19·0	15·0
Walnut oil	20·0	16·5
Castor oil	13·0	3·0
Palm oil	47·8	42·7
Cacao butter	52·0	51·0
Nutmeg butter	42·5	40·0
Cocoanut oil	24·6	20.4
Laurel oil	27·0	22·0
Shea butter	39·5	38·0
Tallow	45·0	43-50
Wool fat	41·8	40·0
Butterine	42	39·8

Determination of the Melting and Congealing Points of Oils and Fats.—It is important that oils used for lubrication should not have very high congealing or solidifying points. The temperature at which an oil congeals may be found by half filling a test tube, 6 in. by ½ in., with the oil, placing a thermometer therein, and introducing the whole into a mixture of equal parts of salt and ice; or if ice be not available, into a mixture of fifteen parts of sodic sulphate, and ten parts of 5 E HCl. The oil is stirred with the thermometer until on inclining the test tube the oil ceases to run; the temperature at which this takes place is taken as the congealing point. On taking the test tube out of the mixture the temperature at which the oil melts could be obtained. It is important that the sample should be free from water and suspended matter before obtaining the melting or congealing point, since these interfere with the accuracy of the results. To obtain the melting and congealing points of oils, solid or semi-solid at the ordinary temperature, a small tall beaker is nearly filled with mercury, and placed in a bath of cold water, the surrounding water being slightly higher than the level of the mercury. A thermometer is suspended from a support, the bulb of which is immersed in the beaker of mercury; a little of the fat—about the size of a pin's head—is placed on the mercury by means of a pair of glass rods drawn out to fine points, and a small flame placed under the water bath, regulated so that the temperature of the mercury rises about 0·5° C. per minute. Note the temperature at which the sample begins to melt, and also the temperature at which it is perfectly fluid. By taking the lamp away it could be observed, as the temperature sinks, at what point the sample congeals.

Good mineral oils do not congeal at the freezing point of water; the heavier varieties congeal at about — 6° to — 8° C., and the lighter kinds remaining fluid at — 16° to — 18° C.

The following results were obtained at the U.S. Navy Yard, Brooklyn, in 1870, with various oils:—

	Thickens. ° C.	Flow ceases. ° C.	Solid. ° C.	Sp. gr.
Sperm . .	1	— 3	— 9	·761
Olive . .	10	— 8	— 14	·933
Tallow . .	— 1	— 4	— 8	·795
„ . .	21	16	6	·993
Lard . .	7	0	— 6	·959

The following are congealing points of the more important oils:—

	° C.
Rape oil, about	— 8
Cottonseed oil, about	— 3
Sunflower „ „	— 14
Linseed „ „	— 24
Hempseed „ „	— 20
Castor „ „	— 18
Poppyseed „ „	— 18
Neatsfoot „ „	— 4

The following are melting points of a few fats, solid at the ordinary temperature:—

		°C.
Cacao butter,	about	32
Palm oil	,,	35
Cocoanut oil	,,	26
Tallow	,,	43
Butter	,,	34
Butterine	,,	38
Beeswax	,,	63
Spermaceti	,,	46

Loss on Evaporation, and Gumming Properties of Oils.—To determine the loss an oil sustains on exposure at elevated temperatures, weigh out about 1 grm. of the oil on a weighed watch glass, allow to stand exposed in a water oven for twelve hours, cool and reweigh, and calculate the loss or gain, as the case may be; observe if the residue is at all sticky or gummy. If the oil be required for light machinery, exposure for twelve hours at 60° C. instead of 100° will be found suitable. Good mineral oils, intended for heavy machinery, cylinders, &c., rarely lose more than 1 per cent. on exposure for twelve hours at 100° C., while mineral oils intended for light machinery rarely exceed a loss of 0·5 per cent. on exposure for twelve hours at 60° C. Many vegetable oils of the "drying" class, such as linseed, hempseed, cottonseed, &c., increase in weight on exposure for twelve hours at 100° C., owing to the absorption of oxygen from the air.

Estimation of Suspended Matter.—Oils intended for lubrication should only contain, at most, a mere trace of suspended matter. Some mineral oils are so dark and thick that a fair quantity of suspended or mineral matter would escape observation in the ordinary way. To determine the proportion that may be present, weigh out about 10 grms. of the sample into a beaker, dilute with 50 cc. of methylated ether, filter off any suspended matter through a weighed filter paper, wash the filter till free from oil with ether, dry and weigh. Increase = suspended matter both mineral and organic. If the amount be large, it would be wise to ignite the filter and its contents carefully in a weighed crucible, and estimate the residual mineral matter. The loss on the former weight would represent suspended organic matter.

Characteristics of Good Lubricating Oils.—The following should be the ideal characteristics of good lubricating oils, according to Spon :—

(1) A mineral oil flashing below 150° C. is unsafe.

(2) A mineral oil losing more than 5 per cent. in ten hours at 15° to 20° C. is inadmissible, as the evaporation creates a gum, or leaves the bearing dry.

(3) The most fluid oil, that will remain in its place, fulfilling other conditions, is the best for all light bearings at high speeds.

(4) The best oil is that which has the greatest adhesion to metallic surfaces, and the least cohesion in its own particles ; in this respect fine mineral oils stand first, sperm oils second, neatsfoot oil third, and lard oil fourth; consequently the finest mineral oils are best for light bearings and high velocities. The best animal oil to give body to fine mineral oils is sperm oil ; lard and neatsfoot oils may replace sperm oil when greater tenacity is required.

(5) The best mineral oil for steam cylinders is one having a density of ·893, and a flashing point of 360° C.

(6) The best mineral oil for heavy machinery has a density of ·880, and a flashing point of 269° C.

(7) The best mineral oil for light bearings and high velocities has a density of ·871, and a flashing point of 262° C.

(8) Mineral oils alone are not suited for very heavy machinery, on account of their want of body, but well purified animal oils are applicable to the heaviest machinery.

(9) Olive oil stands first among vegetable oils, as it can be purified without the aid of mineral acids. The other vegetable oils which, though far inferior to olive oil, are admissible as lubricants are—in their order of merit—sesamé, earth-nut, rape and colza, and cottonseed oils.

(10) No oil is admissible which has been purified by means of mineral acids.

The following list of oils, by Duggan, are placed in the order of their usual commercial monetary value:—

(1)		Olive oil.
(2)		Sperm oil.
(3)		Neatsfoot oil.
(4)	(*a*)	Bottlenose oil.
	(*b*)	Lard oil.
	(*c*)	Castor oil.
(7)		Cod oil.
(8)	(*a*)	Arachis oil.
	(*b*)	Sesamé oil.
	(*c*)	Poppyseed oil.
(11)		Colza and Rape oil.
(12)		Seal oil.
(13)		Nigerseed oil.
(14)		Linseed oil.
(15)		Whale oil.
(16)		Cottonseed oil.
(17)		Menhaden oil.
(18)		Japan Fish oil.
(19)		Mineral oils.
(20)		Resin oil.

Foreign Admixtures in Oils.—The following is a useful table, by Allen, for the examination of oils containing foreign admixtures:—

SCHEME FOR ANALYSING OILS CONTAINING FOREIGN ADMIXTURES.

<table>
<tr><td colspan="6">From 5 to 10 grms. of the sample (previously melted if necessary) is passed through a filter, unless clear.</td></tr>
<tr><td rowspan="5"><i>Residue</i> may contain <i>salt</i>, <i>curd</i>, <i>water</i>, <i>sand</i>, and insoluble matter generally. It may be washed with ether, dried, and weighed; then ignited, and weighed again, the difference being the organic matter.</td><td colspan="5"><i>The clear oil</i> is agitated in a tapped separator with water and ether, or recently distilled carbon disulphide The aqueous liquid is separated, and the oil solution again shaken with water, if the previous treatment was found to remove anything.</td></tr>
<tr><td rowspan="4"><i>Aqueous liquid</i> may contain soaps of the light metals. It is evaporated to dryness at 100° C., and the residue weighed and further analysed if desired.</td><td colspan="4"><i>Oil solution.</i> Agitate with 5 E sulphuric acid and separate. Wash residual oil repeatedly by agitation with water till the aqueous liquid no longer reddens litmus paper.</td></tr>
<tr><td rowspan="3"><i>Acid liquid</i> may contain sulphates of aluminium and heavy metals previously existent as soaps.</td><td colspan="3"><i>Solution of oil in ether or carbon disulphide.</i> Distil off the solvent and heat the residual oil with alcohol and a few drops of solution of phenolphthalein. Then add <i>standard</i> E soda cautiously, agitating the whole between each addition until a pink colour is obtained, which remains after shaking. Note the volume of soda solution required, as the quantity used is a measure of the free, fatty, and resin acids, and will determine the necessity of looking further into their nature. Separate any undissolved oil, dilute the alcoholic liquid, evaporate off the alcohol at a gentle heat and agitate with petroleum spirit, separate, evaporate off solvent, and add any separated oil to the main portion.</td></tr>
<tr><td rowspan="2"><i>Aqueous liquid.</i> Add 10 E HCl and agitate with ether or carbon disulphide; evaporate the ethereal layer at 100°, and weigh residue, representing mixed, free, fatty, and resin acids of original sample, and if soaps of aluminium or heavy metals were present, of the fatty acids resulting from their decomposition.</td><td colspan="2"><i>Oil.</i> Saponify with 1½ E alcoholic potash solution. Boil off alcohol, dissolve soap in warm water, and agitate cooled solution with ether. Separate and agitate aqueous liquid a second and third time with ether.</td></tr>
<tr><td><i>Aqueous liquid</i> contains the glycerine and the soap formed by saponification of the neutral fixed oil of the sample. If the solution be treated with 10 E HCl and agitated with ether, the weight of the fatty acids left on evaporating the ethereal layer, multiplied by 1·055 will give approximately the weight of the neutral fixed oil.</td><td><i>Ethereal liquid</i> distilled at 100° C., and the last traces of the solvent removed by a current of air or other means, leaves the hydrocarbon oils in a state fit for identification and farther examination.</td></tr>
</table>

MINERAL ILLUMINATING OILS.

The mineral illuminating oils of commerce are obtained by distilling (1) the natural crude petroleum oils found as springs and subterranean reservoirs in close proximity to deposits of bitumen, bituminous coals, &c., in America, South Russia, India, Persia, &c., and (2) the crude oil obtained by the distillation of bituminous shale. The oil fields of Pennsylvania supply most of the burning oils of commerce.

There are many fancy names given to the various fractions of the distillation of crude petroleum and shale oils sold as burning oils, such as "kerosene," "photogene," "petroline," "liquid gas," "beacon oil," "aurora oil," "astral oil," &c.

The following table by Redwood* shows the percentage of burning oils obtained from crude petroleums of various localities:—

Locality.	Specific gravity.	Yield of Commercial Products.			
		Naphtha	Burning oil.		Lubricating oil.
		per cent	per cent	sp. gr.	per cent.
Persia	·777	1.4	87·5	—	—
East India	·821	3·6	62·5	·800	32·0
Burmah, mud volcano, Kyouk Phyou	·818	nil	55·7	·800	31·3
„ native pits, Mimbyin	·866	„	15·1	·810	65·9
„ Western Barangah	·888	„	7·2	·815	89·3
„ Eastern Barangah	·835	2·5	66·1	·810	27·3
Assam	·933	nil	nil	—	94·2
India	·935	„	20·0	·805	60·0
Russia	·836	20·0	40·0	—	37·5
„	·942	nil	nil	—	90·0
Hanover	·843	10·0	60·0	·812	27·5
South America	·852	nil	50·0	·808	45·0
„	·900	„	nil	—	91·5
New Zealand	·828	„	60·0	·808	38·0
Italy, near Milan	·787	45·0	45·0	·806	5·4
United States, Wyoming	·910	2·5	27·5	—	57·5
„ „	·945	nil	10·0	—	72·5
Scotch Shale Oil	·870	6	36	·810	18·0

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* "Journ. Soc. Arts," xxxiv., 823, 878.

Examination of Burning Oils.—One of the most important points to be determined in the selection of mineral oils for burning purposes is the temperature at which the vapour evolved from the oil takes fire on the application of a flame, and is termed the "flashing point." The minimum flashing point by the close test about to be described, has been fixed by the Petroleum Act of 1879 to be 73 degrees of Fahrenheit's thermometer.

The following is a sketch of the apparatus and *modus operandi* of the process for the determination of the flashing points of light mineral oils as devised by Sir Frederick Abel, and set forth in Schedule I. of the Petroleum Act of 1879:—

Specification of the Test Apparatus (Fig. 60).—The oil cup consists of a cylindrical vessel 2 in. in diameter and $2\frac{2}{10}$ in. in height (internal), with outward projecting rim $\frac{5}{10}$ in. wide, $\frac{3}{8}$ in. from the top and $1\frac{7}{8}$ in. from the bottom of the cup. It is made of gun-metal or brass (17 B.W.G.) tinned inside. A bracket consisting of a short stout piece of wire bent upwards, and terminating in a point, is fixed to the inside of the cup to serve as a gauge. The distance of the point from the bottom of the cup is $1\frac{1}{2}$ in. The cup is provided with a close-fitting overlapping cover made of brass (22 B.W.G.), which holds the thermometer and test lamp. The latter is suspended from two supports from the side by means of trunnions, upon which it may be made to oscillate; it is provided with a spout, the mouth of which is $\frac{1}{16}$ in. in diameter. The socket which is to hold the thermometer is fixed at such an angle, and its length so adjusted, that the bulb of the thermometer when inserted to its full depth shall be $1\frac{1}{2}$ in. below the centre of the lid.

The cover is provided with three square holes, one in the centre $\frac{6}{10}$ in. × $\frac{4}{10}$ in., and two smaller ones $\frac{2}{10}$ in. × $\frac{2}{10}$ in. close to the sides and opposite each other. These three holes may be closed and uncovered by means of a slide moving in grooves, and having perforations corresponding to those on the lid.

In moving the slide so as to uncover the holes, the oscillating lamp is caught by a pin fixed in the slide and tilted in such a way as to bring the end of the spout just below the surface of the lid. Upon the slide being pushed back so as to cover the holes, the lamp returns to its original position.

Upon the cover, in front of and in line with the mouth of the lamp, is fixed a white bead, the dimensions of which represent the size of the test flame to be used.

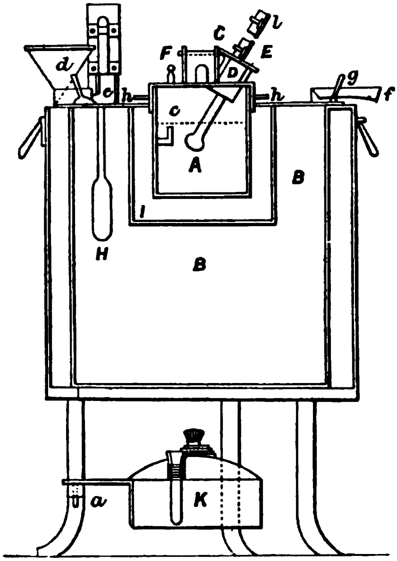

FIG 60.—SIR F. ABEL'S FLASH-POINT APPARATUS.

B Water bath.
A Oil cup.
C Gauge.
E Thermometer.
G Oscillating lamp.
I Air chamber.
H Thermometer.
K Spirit lamp.
F Flame gauge.
d Funnel.

The bath or heated vessel consists of two flat-bottomed copper cylinders (24 B.W.G.), an inner one of 3 in. in diameter and 2½ in. in height, and an outer one 5½ in. in diameter and 5¾ in. in height; they are soldered to a circular copper plate (20 B.W.G.) perforated in the centre, which forms the top of the bath in such a manner as to enclose the space between the two cylinders, but leaving access to the inner cylinder. The top of the bath projects both outwards and inwards about

⅜ in., that is, its diameter is about 6/8 in. greater than that of the body of the bath, while the diameter of the circular opening in the centre is about the same amount less than that of the inner copper cylinder. To the inner projection of the top is fastened, by six small screws, a flat ring of ebonite, the screws being sunk below the surface of the ebonite to avoid metallic contact between the bath and the oil cup. The exact distance between the sides and bottom of the bath and of the oil lamp is ½ in. A split socket, similar to that on the cover of the oil cup, but set at a right angle, allows a thermometer to be inserted into the space between the two cylinders. The bath is further provided with a funnel, an overflow pipe, and two loop handles. The bath rests upon a cast-iron tripod stand, to the ring of which is attached a copper cylinder or jacket (24 B.W.G.), flanged at the top, and of such dimensions that the bath while firmly resting on the iron ring just touches with its projecting top the inward turned flange. The diameter of this outer jacket is 6½ in. One of the three legs of the stand serves as a support for the spirit lamp, attached to it by means of a small swing bracket. The distance of the wick-holder from the bottom of the bath is 1 in. Two thermometers are provided with the apparatus, the one for ascertaining the temperature of the bath, the other for determining the flashing point.

The thermometer for ascertaining the temperature of the water has a long bulb, and a space at the top; its range is from about 90° to 190° F. The scale (in degrees of Fahrenheit) is marked on an ivory back, fastened to the tube in the usual way. It is fitted with a metal collar fitting the socket, and a part of the tube below the scale should have a length of about 3½ in., measured from the lower end of the scale to the end of the bulb. The thermometer for ascertaining the temperature of the oil is fitted with collar and ivory scale in a similar manner to the one described. It has a round bulb, a space at the top, and ranges from about 50° to 150° F. It measures from end of ivory back to bulb 2½ in.

Note.—A model apparatus is deposited at the Weights and Measures Department of the Board of Trade.

Directions for Applying the Flashing Test.—(1) The test apparatus is to be placed for use in a position where it is not exposed to currents of air or draught.

(3) The test lamp is prepared for use by fitting it with a piece of flat-plaited candle wick, and filling it with colza or rape oil to the lower edge of the opening of the spout or wick tube. The lamp is trimmed so that when lighted it gives a flame of about 0·15 of an inch in diameter, and this size of flame, which is represented by the projecting white bead on the cover of the oil cup, is readily maintained by simple manipulation from time to time with a small wire trimmer. When gas is available it may be conveniently used in place of the little oil lamp, and for this purpose a test flame arrangement for use with gas may be substituted for the lamp.

(2) The heating vessel or water bath is filled by pouring water into the funnel until it begins to flow out at the spout of the vessel. The temperature of the water at the commencement of the test is to be about 130° F., and this is attained in the first instance either by mixing hot and cold water in the bath, or in a vessel from which the bath is filled until the thermometer which is provided for testing the temperature of the water gives the proper indication; or by heating the water with the spirit lamp (which is attached to the stand of the apparatus) until the required temperature is indicated. If the water has been heated too highly, it is easily reduced to 130° F. by pouring in cold water little by little (to replace a portion of the warm water) until the thermometer gives the proper reading. When the test has been completed, this water-bath is again raised to 130° F. by placing the lamp underneath, and the result is readily obtained while the petroleum cup is being emptied, cooled, and refilled with a fresh sample to be tested. The lamp is then turned on its swivel from under the apparatus, and the next test is proceeded with.

(4) The bath having been raised to the proper temperature,

the oil to be tested is introduced into the pretroleum cup, being poured in slowly until the level of the liquid just reaches the point of the gauge which is fixed in the cup. In warm weather the temperature of the room in which the samples to be tested have been kept should be observed in the first instance, and if it exceeds 65° F. the samples to be tested should be cooled down (to about 60° F.) by immersing the bottles containing them in cold water, or by any other convenient method. The lid of the cup with the slide closed is then put on, and the cup is placed into the bath or heating vessel. The thermometer in the lid of the cup has been adjusted so as to have its bulb just immersed in the liquid, and its position is not under any circumstances to be altered. When the cup has been placed in the proper position, the scale of the thermometer faces the operator.

(5) The test lamp is then placed in position upon the lid of the cup; the head line or pendulum, which has been fixed in a convenient position in front of the operator, is set in motion; and the rise of the thermometer in the petroleum cup is watched. When the temperature has reached about 60° F., the operation of testing is to be commenced, the test flame being applied once for every rise of 1° F. in the following manner:—

The slide is slowly drawn open while the pendulum performs three oscillations, and is closed during the fourth oscillation.

Note.—If it is desired to employ the test apparatus to determine the flashing points of oils of very low volatility, the mode of proceeding is to be modified as follows:—

The air chamber which surrounds the cup is filled with cold water to a depth of 1½ in., and the heating vessel or water bath is filled as usual, but also with cold water. The lamp is then placed under the apparatus and kept there during the entire operation. If a very heavy oil is being dealt with, the operation may be commenced with water previously heated to 120° F., instead of with cold water.

Pensky-Marten's Flash-point Apparatus.—This apparatus, which is designed for the testing of heavy oils, consists of an oil cup with cover fitted with thermometer and stirrer, and an air bath. The bath may be heated by a gas flame or spirit lamp, and the slow and regular heating of the oil is ensured by the jacket of air that surrounds the cup.

The determination of the flashing point is effected by rotating the vertical spindle by means of the milled head (G), *vide* Fig. 61, whereby the sliding cover is opened, and simultaneously a small flame burning at the movable jet is brought to the opening.

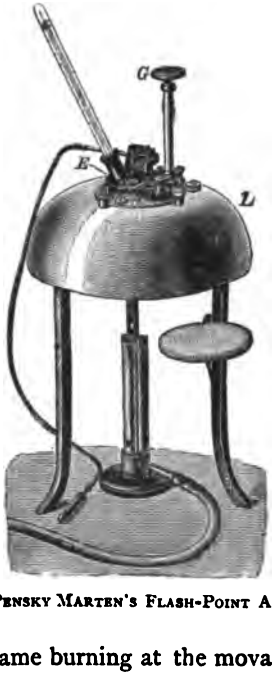

FIG. 61.—PENSKY MARTEN'S FLASH-POINT APPARATUS.

This is repeated at intervals during the rise of temperature until a flash occurs in the space above the oil, and the temperature at which this occurs is the flashing point.

The cup is filled to the ring inside with the oil to be tested, and the cover placed in position so that the test-jet is not above either of the two hooks on the collar of the cup. The cup is then placed in the bath by means of the fork provided for that purpose, which ensures steadiness of movement, and prevents the walls of the cup becoming oily above the proper level. The thermometer having been inserted in the socket in the cover, the gas burner is lighted beneath the wire gauze screen, and the rate of heating so adjusted that the temperature of the oil under examination rises 10° F. in each minute. The gas test-jet is then lighted, and is regulated by means of the screw valve until the flame is of the size of a small pea. During the heating of the oil the stirrer is used from time to time. At each rise of 1° of the thermometer, the test flame is applied by rotating the spindle until the flashing point is reached, the use of the stirrer being intermitted while this is done.

If the flashing point of the oil brought under test is known, approximately, the rate of heating may at first be accelerated by using a larger flame and by removing the wire gauze, but the rate must be adjusted to 10° F. per minute at least 20° below the flashing point.

As traces of the more volatile hydrocarbons considerably reduce the flashing point, it is essential that the apparatus during use should be kept free from such. It is also necessary to keep the oil cup perfectly dry, as traces of moisture interfere with the production of the flash. Care must, therefore, be taken that the oil is free from water before testing.

If the heating agent is gas the test-flame is also gas, but where gas is not available the test-flame is fed with rape or mineral oil. The oil flame is regulated by adjusting the wick, the gas-jet by a regulating screw.

Fractional Distillation.—In addition to obtaining the specific gravity and flashing points of mineral burning oils, the amount of distillates, with their specific gravities on fractional distillation, would be a valuable means of judging their qualities. They should yield a comparatively small quantity of distillate below 150° C., and a small percentage of heavy oils as a residuum at 300° C. A good method for the fractional distillation of burning oils is that recommended by Biel:—250 grms. of the oil are weighed into a 500 cc. flask which is covered with brass wire gauze to maintain an even temperature. It is connected to a Glinsky's dephlegmator and a Liebig's condenser. A thermometer is fixed in the dephlegmator, the bulb of which is to be on a level with the exit tube. A small flame is lighted under the flask, and the distillate up to 150° C. is collected in a weighed receiver and weighed. A second weighed receiver is replaced on the condensing tube and the flame enlarged, and the heating continued until practically all has distilled up to a temperature of 270° C., which is weighed. The difference between the weights and the 250 grms. taken, may be considered the "tailings" or heavy oils left in the retort at 270° C.

Biel obtained the following results by the above process with different Russian burning oils:—

	Kerosene.			Pyronaphtha.	
	A.	B.	C.	D.	E.
Specific Gravity . . .	·820	·820	·835	·857	·867
Flash Point ° C. . . .	52·5	35·0	44·5	7·5	94·0
Light Oil (below 150°) .	0·8%	10·0%	6·0%	0·0%	0·0%
Normal Oils (150° to 270°) .	92·0%	76·5%	63·5%	44·5%	30·5%
Heavy Oils (tailings) . .	7·2%	13·5%	30·5%	55·5%	69·5%

The following results were obtained by Biel with American and Russian burning oils :—

	American.		Baku.	
	No. 1.	No. 2.	No. 1.	No. 2.
Specific Gravity	·795	·783	·803	·822
Flash Point ° C.	26	48	26	30
Light Oil (below 150° C.) . .	14·4 %	2·2 %	33·5 %	12·8 %
Normal Oils (150° to 270° C.) .	45·9 %	87·8 %	66·5 %	78·3 %
Heavy Oils (tailings) . . .	39·7 %	10·0 %	..	8·4 %

The following table by Redwood* shows the distinctive differences between Russian and American burning oils (kerosene). Nine fractions of 10 per cent. each were distilled off, and their respective specific gravities taken at 15° C., and also the 10 per cent. of residuum left in the retort :—

Original Sample.	Russian Kerosene. Specific gravity ·822.	American Kerosene. Specific gravity ·803.
1st fraction	·783	·748
2nd ,,	·796	·759
3rd ,,	·803	·778
4th ,,	·814	·792
5th ,,	·827	·802
6th ,,	·831	·812
7th ,,	·837	·822
8th ,,	·838	·831
9th ,,	·846	·838
10th ,, (residue)	·864	·849

In judging the quality of various burning oils for lighting railway carriages, &c., it would be wise to make a practical test under the same conditions as in actual use, noting:—

(1) The intensity of light at start;

(2) The intensity of light towards the end;

(3) The consumption of oil per hour;

(4) The condition of wick at finish;

comparing with what is considered a standard oil under the same conditions.

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* *Jour. Soc. Chem. Ind.*, iv., 76.

COMPARATIVE COST OF PETROLEUM AS AN ILLUMINANT.

The following table by Redwood, shows the comparative cost of a given amount of light from petroleum oil, colza oil, and coal gas, equal to that of a standard sperm candle, burning at the rate of 120 grains per hour for 1,000 hours :—

COST OF 1,000 CANDLE HOURS.

From Petroleum Oil.				From Colza Oil.			From Coal Gas.		
Price per gallon.				Price per gallon.			Price per 1,000 cubic ft.		
d. 9	s. d. 1 0	s. d. 1 3	s. d. 1 6	s. d. 3 0	s. d. 3 6	s d. 4 0	s. d. 3 0	s. d. 3 6	s. d. 4 0
d. 7½	d. 9¾	d. 11¼	s. d. 1 3	s. d. 2 4	s. d. 2 8	s. d. 3 0¾	s. d. 1 3	s. d. 1 5½	s. d. 1 8

From the above figures it will be seen that, taking petroleum at 1s. per gallon and coal gas at 3s. 6d. per 1,000 cubic feet, a given amount of light from the latter costs about twice as much as the former; and taking colza oil at 3s. 6d. per gallon and petroleum oil at 1s. per gallon, it will be seen that petroleum has the advantage threefold for a given amount of light.

Reporting upon the relative cost and efficiency of electricity, gas, and oil as illuminants, the Trinity House Committee conclude, that "for all practical purposes, gas and oil are equal," and that, "for the ordinary necessities of lighthouse illumination, mineral oil is the most suitable and economical illuminant."

THE STORAGE OF PETROLEUM.

Owing to the high degree of inflammability of petroleum oils, it is of importance that great care should be exercised in their storage. The following are some of the best means of safely storing petroleum :—

(1) The oil to be kept in metallic tanks, sunk some distance into the ground, from which quantities can be drawn as required with the aid of a hand pump.

(2) If the ordinary wooden barrels be desired to be kept intact, they should be kept in isolated warehouses of one storey only, built of uninflammable materials.

(3) Weights are attached to the ordinary barrels, which are then sunk in water.

(4) A convenient mode of storing inflammable oils, is to place a vessel similar to a gasholder, mouth downwards into a large tank filled with water, in which the oil can thus be stored over the water, from which quantities can be drawn as required.

Notes on the Construction of Petroleum Lamps.—The Metropolitan Board of Works (now London County Council) have issued the following instructions as to the construction and management of ordinary mineral-oil lamps:—

(A) *Lamps.*

(1) That portion of the wick which is in the oil reservoir should be enclosed in a tube of thin sheet-metal, open at the bottom, or in a cylinder of fine wire gauze, such as is used in miners' safety-lamps (28 meshes to the inch).

(2) The oil reservoir should be of metal rather than china or glass.

(3) The oil reservoir should have no feeding-place or opening, other than the opening into which the upper part of the lamp is screwed.

(4) Every lamp should have a proper extinguishing apparatus.

(5) Every lamp should have a broad and heavy base.

(B) *Wicks.*

(1) Should be soft, and not tightly plaited.

(2) Should be dried at the fire before being put into lamps.

(3) Should be only just long enough to reach the bottom of the oil reservoir.

(4) Should be so wide that they quite fill the wick-holder without having to be squeezed into it.

(C) *Management.*

(1) The reservoir should be quite filled with oil every time before using the lamp.

(2) The lamp should be kept thoroughly clean. All oil should be carefully wiped off, and all charred wick and dirt be removed, before lighting.

(3) When the lamp is lit, the wick should be first turned down and then slowly raised.

(4) Lamps which have no extinguishing apparatus should be put out as follows :—The wick should be turned down until there is only a small flickering flame, and a sharp puff of breath should then be sent *across* the top of the chimney but not *down* it.

(5) Cans or bottles used for oil should be free from water and dirt, and should be kept thoroughly closed.

Instructions for the Use of the Redwood Apparatus for Detecting Inflammable Vapours on Board Petroleum Tank Steamships.*

The complete appliances are shown in the illustrations below—Figs. 62 to 64. They consist of the lamp A, the reservoir of compressed hydrogen B, and the sampling vessel C, in which the sample of air for examination is collected. The lamp is shown in section in Figs. 62 to 64. A is the hydrogen inlet tube with the regulating valve B, and C is the hydrogen jet. D is the inlet tube, for the sample of atmosphere to be tested. The bore of this tube is greatly contracted, and immediately above the point at which this tube enters the base of the lamp is an arrangement of baffles, surmounted by three disks of wire gauze of at least 28 wires per lineal inch, or not less than 784 openings per square inch, the flow of the gaseous mixture to the flame being thus regulated, and passage of flame into the collecting vessel being prevented. The chimney E fits air-tight at the base, but is capable of vertical movement on an inner tube, the front of which is removed. The chimney is partly of metal and partly of glass, the metallic portion being blackened inside, and on the glass window lines corresponding with various heights of flame caps may be marked. The top of the hydrogen jet tube is 10 millimetres (0·4 inch) below the bottom of the window. Attached to the base of the lamp is a telescopic support for a

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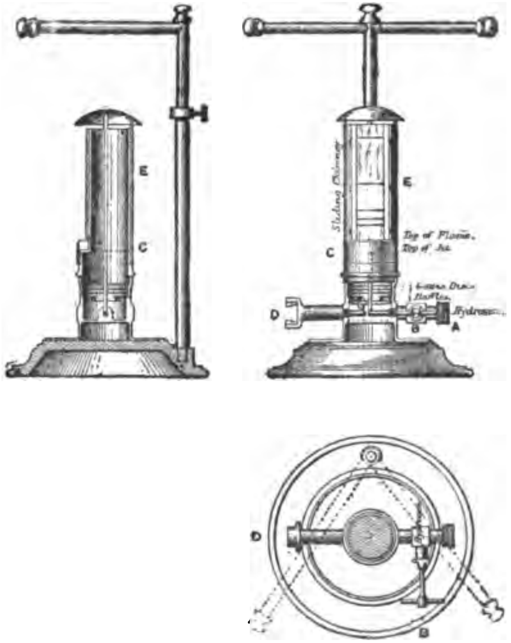
* *Vide* Proc. Inst. Civil Engineers, Vol. cxvi., Part ii.



FIGS. 62, 63, 64.

cloth, which envelops the head of the observer and excludes light when the testing apparatus is used in an undarkened room. The construction of the collecting vessel is shown in section in Fig. 65. A is the compression pump, which is furnished with a metallic spring piston, fitting the pump cylinder without the use of leather or other material, and lubricated with plumbago. Surrounding the pump is an annular space, in which the sample of atmosphere is stored. B is a collar to which may be attached a flexible suction tube of any desired length. C is a cock, to which is attached a copper tube conveying the sample to the test lamp. The bore of this cock is very much reduced. D is a pressure gauge, and E a spring valve lifting at 30 lbs. pressure. F F are hinged brackets, on which the feet of the operator are placed while the pump is being worked. G is a handle, by which the cylinder can be conveniently carried. The capacity of the pump is 14·84 cubic inches, and of the annular space 169·14 cubic inches, thirty double strokes of the pump being required to charge the vessel to a pressure of 30 lbs. per square inch, when it will contain ¼ cubic foot of the atmosphere sampled.

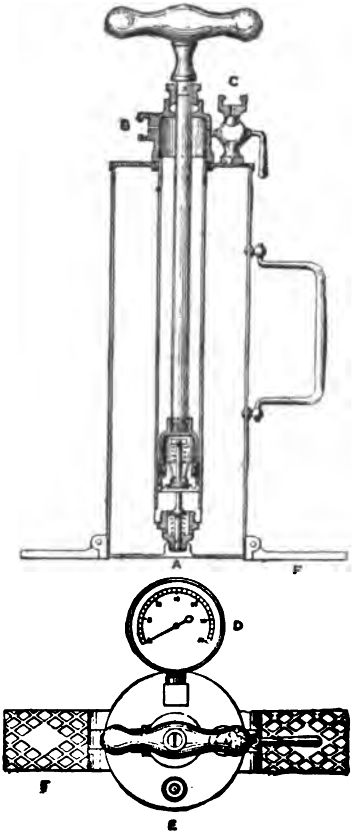


Fig. 65.

In the use of the apparatus, the first step is to connect the hydrogen cylinder with the lamp, taking care that the unions are screwed up gas-tight. The sliding chimney of the lamp being raised about half way, the gas is then cautiously turned on at the cylinder, the regulating valve on the lamp being left open, and a light is applied to the hydrogen jet. The valve on the hydrogen cylinder is then adjusted so as to give a flame rather more than 10 millimetres (0·4 inch) in length, and the lamp chimney pushed down until there is an opening of only about ¼ inch in height at the bottom. This opening is left for the supply of air to the hydrogen flame during the few minutes occupied in the warming of the chimney.

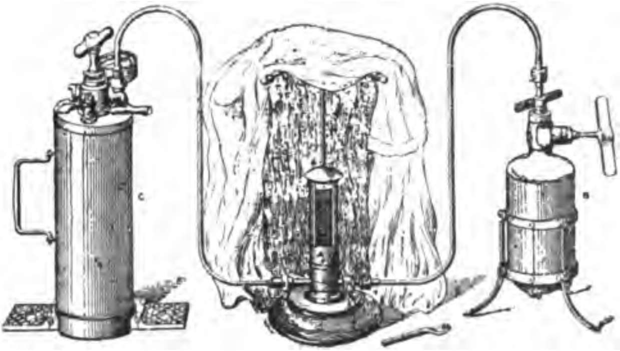


FIG. 66.

As soon as the moisture which at first condensed upon the cold glass has evaporated, the lamp is ready for use, and assuming the collecting vessel to have been already charged with the sample to be tested, and connected with the lamp (see Fig. 66), all that remains is to completely close the sliding chimney of the lamp, adjust the hydrogen flame by means of the regulating valve on the lamp, so that the tip of the flame is only just hidden when the eye of the observer is on a level with the bottom of the window, place his head under a cloth, such as used by photographers, so as to exclude light, and as soon as his eyes have become sufficiently sensitive, turn on the tap of the collecting cylinder, and carefully observe what takes place in the lamp chimney. The tap may at once be turned on fully, as the contraction of the outlet and inlet orifices, already referred to, prevents the sudden rushing out of the contents of the cylinder, and the sample will be gradually delivered into the test lamp during a period of more than two minutes, which is ample time for noting the effect.

The rate of delivery, of course, gradually diminishes, but this is not found to be attended with any inconvenience, the conditions being the same in each experiment. In this way a proportion of vapour, considerably below that which is required even for the production of an inflammable mixture, and still lower than that which is needed to give an explosive atmosphere, may be detected by the formation of a flame cap of greyish-blue colour, which, though faint, is nevertheless easily seen, especially after a little practice. With an increase in the quantity of vapour, the flame cap first becomes much better defined, though it is not greatly augmented in size, and then considerable enlargement of the cap occurs, this condition being arrived at before the atmosphere becomes inflammable.

The Petroleum Act, 1879.—The text of this Act will be found in the Appendix (p. 394).

OILS FOR VARIOUS PURPOSES.

Gas Oils.

Oils obtained from crude petroleum and bituminous shale oil are now largely used in the manufacture of gas for the lighting of railway carriages, &c. The great point in the selection of oils for this purpose, is to ensure uniformity of supplies, when once the right kind of oil has been decided upon that is suited to the plant used.

The following is the specification of the Great Eastern Railway Company for shale gas oil, for use with their Pintsch apparatus :—

"The specific gravity to be not less than ·855 at 60° F.

(= 15·5° C.). When subjected to fractional distillation, free ebullition is to commence at a temperature of about 550° F. (= 287·8° C.), and the first tenth by volume of the distillate is not to exceed ·840 sp. gr. at 60° F. (= 15·5° C.). The succeeding tenths by volume of distillates are to exhibit a fairly uniform rate of increase in density, and the specific gravity of the tenth or final fraction, is not to exceed ·880 at 60° F. (= 15·5° C.)."

The following results were obtained for three samples of shale gas oil supplied by three different firms, in response to inquiries for quotations for gas oil on the basis of the above specification:—

	No. 1.	No. 2.	No. 3.
Specific gravity at 15·5° C. . .	·869	·847	·887
Commencement of free ebullition .	287·8° C.	287·8° C.	(above) 287·8° C.

Results of Distillations:—Specific gravity of fractions at 15·5° C.

No. of fraction.	No. 1.	No. 2.	No. 3.
1	·843	·830	·863
2	·845	·834	·868
3	·848	·837	·869
4	·859	·838	·875
5	·861	·841	·878
6	·866	·843	·880
7	·871	·847	·885
8	·878	·851	·889
9	·879	·856	·893
10	·880	·874	·906

The following are some interesting results obtained by Dr. Macadam on experimenting with Pintsch's and Keith's apparatus:—

BLUE PARAFFIN OIL USED.

	Pintsch's Apparatus.			Keith's Apparatus.		
	A	B	Average.	A	B	Average.
Specific gravity of oil	877·6	878·2	877·9	874·1	877·6	875·9
Flashing point	296°	294°	295°	292°	286°	289°
Firing point	356°	352°	354°	348°	346°	347°
Gas per gallon, cubic feet	90·7	103·4	97	85	84·8	84·9
Illuminating power	62·5	59·1	60·8	63·2	59·5	61·4
Volume of oil flowing into each retort per hour	1·4	1·18	1·29	2·3	1·3	1·8
Gas per retort per hour	126·8	122·5	124·6	197·5	111·9	154·7
Heavy hydrocarbons per cent.	39·2	37·1	38·2	39·9	38·2	39·0
Gas per ton (cubic feet)	23,128	26,356	24,742	21,772	21,671	21,721

Oil of Turpentine ($C_{10}H_{16}$).

Pure oil of turpentine is obtained by distilling the oleo-resinous juice got from the bark, &c., of pine and fir trees, &c., and which yields from 10 to 30 per cent. of turps, leaving a residuum of resin or colophony.

American oil of turpentine has a specific gravity of ·864 to ·870, and commences to boil at about 155° C., and on distillation it completely passes over below 180° C.

Turpentine is often largely adulterated with petroleum products or " turpentine substitutes."

The writer recently examined a sample of supposed genuine turpentine, and found it to be wholly American kerosene. A good practical test of the drying properties of turpentine, is to drop a little of the sample on a piece of writing paper and allow it to evaporate at the ordinary temperature; genuine turpentine completely evaporates in a short time without leaving a trace of a greasy stain, while if kerosene, &c., be present, a more or less permanent greasy stain will be produced which, on gently heating, will give an odour characteristic of its nature.

In making an assay of a sample of turpentine, the following are the most important points to be ascertained :—

(1) *The Specific Gravity.*—Genuine turpentine should have a specific gravity of ·864 to ·870 ; if it is much lower than this, it is almost sure to contain foreign hydrocarbons.

Resin spirit has a sp. gr. of	·856 to ·880.
Shale naphtha . .	·700 „ ·750.
Coal tar solvent naphtha	·860 „ ·876.
Petroleum naphtha .	·700 „ ·745.

(2) *The Flashing Point.*—American oil of turpentine flashes at about 34° C., and if any petroleum spirit or naphtha be present it would be considerably lower.

(3) *The Boiling Point.*—The range of the boiling point of genuine turps would be from 155° to 180° C. ; while if petroleum products were present, the range would be considerably higher.

(4) *Distillation.*—Distil 100 cc. of the sample in a 150 cc. retort, to 180° C., and collect the distillate in a graduated tube. With good turps at least 97 per cent. should come over at this temperature, and the residue in the retort would be oxidized products of the turpentine ; if there is any considerable residue left in the retort at this temperature, it points to adulteration with hydrocarbon of higher boiling point. Obtain its specific gravity with a Sprengel tube. By distillation in a current of steam pure turps leave a small residue of about 0·5 per cent. ; genuine old samples, however, leave as much as 2 per cent. residue, owing to the turpentine having absorbed atmospheric oxygen, forming resinous substances. If any petroleum is present it is unvolatilized, and may be recognised by its low specific gravity as compared with that of turpentine.

For the determination of petroleum in turpentine the following method* will be found to give fair results :—

A balloon flask of 750 cc. capacity is fitted with a two-hole cork stopper. Through one hole is inserted the tube of a glass-stoppered drop funnel, having a capacity of 100 cc. The flask is also connected with an inverted condenser. About 300 cc. fuming nitric acid of sp. gr. 1·4 are placed in the flask, and 100 cc. of the turpentine to be tested are measured into the drop funnel. The flask is surrounded by cold water, and the turpentine is allowed to drop slowly into the nitric acid. As each drop strikes the acid violent action takes place, with evolution of red fumes. It is well to shake the flask occasionally during the operation. When the turpentine has all passed into the flask, the apparatus is allowed to stand until all action is over. The contents of the flask are transferred to a large separating funnel, and treated with successive portions of hot water. In this way all the products resulting from the action of the acid on the terpenes are removed, while any petroleum paraffins remain insoluble in water, and can readily be separated and measured.

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* Vide *Oil and Colourman's Journal*, Dec., 1890.

Having procured some turpentine known to be free from petroleum, mixtures of the pure turpentine with samples of refined petroleum of various boiling points were prepared.

In order that judgment in experimenting might not be influenced by knowing the quantities of material used, the proportions employed in the mixtures were withheld from the operator.

Ten mixtures were analysed, with the following results :—

Pure turpentine used.	Petroleum used.	Boiling point of Petroleum.	Petroleum found by method described.
cc.	cc.	deg.	cc.
65	35	about 250	34·1
80	20	250	18·9
70	30	200	29
80	20	200	18·5
90	10	200	8·9
80	20	100	17·8
70	30	100	28·4
85	15	100	13·5
80	20	75	17·9
70	30	75	28

It will be noticed that the sum of the amounts of petroleum and turpentine used would be 100 cc. in each case, so that the number of cubic centimetres of petroleum found would also express the percentage of adulterant. It appears from the above results that the petroleum fractions, which boil at about 250°, are least affected by the fuming nitric acid, while the low-boiling fractions are affected the most.

But in order to keep the specific gravity of the turpentine where it should be, the higher boiling petroleum fractions must be used in the adulteration—a fact which is favourable for the application of this method in the analysis of such mixtures.

Rape Oil.

Rape oil or *Colza oil* is obtained from the seeds of various species of *Brassica*, chiefly those of *B. campestris* and *B. napus*. The principal applications of rape oil are for illuminating purposes, lubrication, and in the manufacture of india-rubber. It has a brownish-green or yellowish-brown colour, and a characteristic odour. The specific gravity of genuine rape oil ranges between ·913 and ·916 at 15·5° C.; if the density of a sample of rape oil be above ·916 it is highly probable that it is adulterated with oils of higher density and less value, such as the drying oils, of cottonseed, linseed, sunflower, hempseed, &c., whose specific gravities range between ·920 and ·937. When rape oil is intended for lubrication, the presence of drying oils is highly objectionable, since they increase the gumming properties of the oil. A useful test for the drying properties of rape oil, is to expose a thin film of the oil on a glass plate at a temperature of 100° C. for twenty-four hours by the side of a sample of genuine rape, frequently noting the condition of the samples by touching them with the finger. Genuine rape remains comparatively fluid for a few days; but if any drying oil be present it will assume the consistency of a jelly, or in some cases become almost as hard as a resin. Another useful test for the drying properties of rape oil is the Elaïden test described on page 279, under "Various Tests."

The viscosity of the sample at 15·5° C. is another valuable point in judging of its quality, 50 cc. of genuine rape oil taking about 535 seconds to run out of Redwood's viscosimeter at 15·5° C. See table on page 243. Maumené's temperature reaction, described on page 279, would be useful in confirming the presence of some foreign seed oils, the average rise of temperature for genuine rape being about 60° C. by this test.

In addition to the above tests apply the following:—

(1) Ascertain the melting and congealing points of the total fatty acids, as directed on page 254. The fatty acids of good rape oil melt at about 20° C., and congeal at about 17° C.

(2) Determine the percentage of free fatty acids, as described on page 248. The proportion of free fatty acids in good rape oil varies from 0·5 to 4 per cent. Excess of free fatty acids is undesirable when the oil is intended for lubrication, owing to their action on bearings, &c., forming hard metal soaps; and when such an oil is used for illuminating purposes it burns with a smoky flame and chars the wick. If required for soap-making the presence of free fatty acids is less objectionable, since the acids are neutralized in the process.

(3) Test for free mineral acids. The presence of free mineral acids is very objectionable, if the oil is proposed to be used for lubrication. Traces of free sulphuric acid are often found in rape oils, owing to its incomplete elimination after refining.

(4.) Test for mineral oils as directed on page 251.

Olive Oil.

Olive oil, *salad oil*, or *sweet oil* is obtained from the fruit of the *Olea Europæa* by compression, or by extracting with bisulphide of carbon, in which it is soluble. It should be of a greenish-yellow colour, without odour, and of an agreeable taste, and non-drying. Its principal uses are for cooking, wool-dressing, lubricating, medicine, and soap manufacture. Its specific gravity ranges between ·914 and ·918. At a temperature of 0° C. it congeals, which on compression yields one-third of its weight of solid fat (Palmitin and Stearin), melting at from 20° to 28° C.; the two-thirds of the fluid portion (olein), remain liquid at from — 4° to — 10° C. Olive oil, when long exposed, assumes an objectionable rancid odour, owing to the incomplete elimination of mucilaginous matters. In judging of the quality of a sample of olive oil obtain the following points, as described under their respective heads, referring to the various tables to see how near to, or how far from, the genuine article it corresponds:—

(1) Specific gravity at 15·5° C.
(2) Temperature at which fat is deposited.
(3) The percentage of free fatty acids (average should be 5 to 8 per cent).
(4) Maumené's temperature reaction.
(5) Test for mineral oils.
(6) Test for fish oils.
(7) The drying properties, Elaïden test.
(8) Mineral acids.
(9) The viscosity at 15·5° C.
(10) The odour on heating.
(11) Taste.
(12) Acid tests.

Linseed Oil.

This well-known and useful oil is obtained from the seeds of the flax plant—*Linum usitatissimum*—principally grown in India and Russia. It is largely used for paints and varnishes, and in the manufacture of oil-cloth, rick-covers, and soft soap. It is used in four states, according to the purpose for which it is to be applied, viz.:—the raw, refined, artists', and the boiled oil. The colour of the raw oil is yellowish-brown, and its specific gravity varies from ·930 to ·937. At a temperature of — 18° C. a little fat separates out, and it becomes perfectly congealed at — 27° C. The most valuable characteristic of linseed oil is the facility with which it absorbs atmospheric oxygen, with the formation of a tough, resinous-looking substance, materially differing in properties from the original oil. The drying properties of linseed oil are increased after it has been heated to about 180° C., with a current of air passing through, and the heat continued until the oil is seemingly in a state of ebullition owing to the liberation of decomposition products. The addition of drying agents, such as sugar of lead, oxide of manganese, litharge, iron oxide, &c., during the heating, increases the drying properties of the finished or "boiled" oil, owing to their acting as carriers of oxygen.

In judging of the quality of a sample of linseed oil, its drying property is one of the most important points to be determined, and may be ascertained by smearing a glass plate with the sample, allowing it to be exposed in a water oven, and noting how long it takes to dry, and the nature of the residuum left as compared with that of a standard sample. If it be desired to ascertain if any driers have been added, and their nature, gradually ignite about 50 grms. of the sample in a large porcelain crucible, and make a qualitative analysis of the residue.

With regard to other tests of its quality, apply those given under the head of olive oil.

Cottonseed Oil.

This is a drying oil obtained from the *Gossypium barbadense*, and is largely used to adulterate rape oil, olive oil, and other oils. It is also used in cooking, soap-making, and in the manufacture of butterine. The specific gravity of cottonseed oil ranges from ·922 to ·930, and solidifies at from 1° C to 4° C. The melting point of the fatty acids from cottonseed oil is so high as 38° C. It appears that cottonseed oil is not likely to be adulterated itself, but a knowledge of the properties of the oil is of great importance, in ascertaining its presence in other oils. By applying the tests given under those of olive oil, cottonseed oil could be approximately estimated in a mixture.

VARIOUS TESTS FOR OILS.

Maumené's Temperature Reaction of Oils.—The principle of this test depends upon the fact that when 36 E sulphuric acid is mixed with a fatty oil, decomposition takes place, with considerable rise in the temperature, which varies with different oils. The following is the method of applying the test:—50 grms. of the oil are weighed into a 200 cc. tall beaker, and packed with cotton wool into a litre beaker. The temperature of the oil is next observed, and 10 cc. of 36 E H_2SO_4, at the same temperature, gradually run in from a pipette or burette, and should occupy about 60 seconds to run out, the oil being thoroughly stirred with the thermometer the whole time; continue stirring after the addition of the acid, and note the highest temperature the thermometer indicates; on subtracting from this, the initial temperature, the increase in temperature will be obtained. The following are the results obtained by Maumené by the above process with various oils:—

	Rise in Temperature °C.
Olive oil	42
Almond oil	52-54
Rape ,,	57-58
Arachis ,,	67
Beechnut oil	65
Sesamé ,,	68
Poppyseed oil	74
Hempseed ,,	98
Walnut ,,	101
Linseed ,,	103
Castor ,,	47
Tallow ,,	41-44
Horsefoot ,,	51
Cod liver ,,	102-103
Skate liver ,,	102

Poutet's Elaïdin Test.—This is a very useful test for detecting drying oils mixed with non-drying oils and *vice versâ*.

It depends upon the formation of elaïdin (an isomeric modification of olein, and which is solid at the ordinary temperature), when a non-drying oil is subjected to the action of nitrous acid, the drying oils, which consist largely of linolein, being not so affected. The following is the manner in which the test is performed:—Measure out 50 cc. of the oil into a wide-mouthed stoppered bottle of about 100 cc. capacity, and pour on to the oil 2 cc. of a cold freshly-made solution of nitrate of mercury, prepared by dissolving 13 grms. of mercury in 12 cc. of 16 E nitric acid, the beaker in which it is dissolved being kept in cold water to retain nitrous fumes. Agitate the mixture, and allow to stand in an air bath, regulated to 25° C., for twenty-four hours, with occasional shaking, together with a pure standard sample of a similar oil. Note the length of time that solidification takes place, also the consistency of the mass after standing for the twenty-four hours. The drying oils, such as linseed, hempseed, &c., remain fluid, while the non-drying oils, such as olive, tallow oil, almond, sperm, yield hard products; and oils of the intermediate class, such as rape, neatsfoot, sperm, cottonseed, whale, &c., leave a mass more or less of the consistency of treacle.

Sulphuric Acid Test.—This is the best of the various colour tests for oils recommended by Chateau. There are various methods of applying it, the most suitable being that recommended by Allen,* who adds one or two drops of 36 E H_2SO_4 in the centre of twenty drops of the oil, and notes the colour produced before and after stirring, when the following colours are produced with various oils:—

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* *Vide* "Comcl. Org. Analysis," vol. ii., p. 59.

Oil.	One or two drops of 36 E H_2SO_4 to twenty of the oil.	
	Before Stirring.	After Stirring.
Vegetable Oils.		
Olive oil	Yellow, green, or pale brown.	Light brown or olive-green.
Almond oil . . .	Colourless or yellow.	Dark yellow, olive, or brown.
Earthnut oil . . .	Greyish yellow to orange.	Greenish or reddish brown.
Rape oil (crude). .	Green with brown rings.	Bright green, turning brownish.
,, ,, (refined) .	Yellow with red or brown rings.	Brown.
Mustard oil . . .	Dark yellow with orange streaks.	Reddish brown.
Cottonseed oil (crude)	Very bright red.	Dark red, nearly black.
,, ,, (refined)	Reddish brown.	Dark reddish-brown.
Nigerseed oil . . .	Yellow with brown clot.	Reddish or greenish-brown.
Poppyseed oil . .	Yellow spot with orange streaks.	Olive or reddish-brown.
Linseed oil (raw) .	Hard brown or greenish-brown clot.	Mottled dark brown.
,, ,, (boiled)	Hard brown clot.	Mottled dark brown.
Castor oil	Yellow to pale brown.	Nearly colourless or pale brown.
Animal Oils.		
Lard oil	Greenish-yellow or brownish with brown streaks.	Mottled or dirty brown.
Tallow oil	Yellow spot with pink streaks.	Orange red.
Whale oil	Red, turning violet.	Brownish-red, turning brown or black.
Seal oil	Orange spot with purple streaks.	Brownish-red, changing to mottled brown.
Cod liver oil . . .	Dark red spot with purple streaks.	Purple, changing to dark brown.
Sperm oil	Pure brown spot with faint yellow ring.	Purple, changing to reddish or dark brown.
Hydrocarbon Oils.		
Petroleum lubricating oil.	Brown.	Dark brown, with blue fluorescence.
Shale lubricating oil	Dark reddish brown.	Reddish brown, with blue fluorescence.
Resin oil (brown) .	Bright mahogany brown.	Dark brown, with purple fluorescence.
,, ,, (pale) . .	Mahogany brown.	Red brown, with purple fluorescence.

Massie's Nitric Acid Colour Test.—Mix 3 cc. of the oil for two minutes with 1 cc. of pure 16 E nitric acid, and observe the colour of the separated oil. The following are the colours produced with various oils by this method:—

Olive oil	Colourless, yellow or greenish.
Almond oil	Colourless or slightly greenish.
Arachis "	Reddish.
Peach-kernel oil	Immediate red liniment.
Rape oil	Reddish or orange.
Sesamé oil	Yellowish or orange.
Cottonseed oil	Brown or brownish-red.
Linseed "	Red or orange.
Poppyseed "	Reddish.
Hempseed "	Brownish-red.
Castor "	Yellowish or orange.

Tests for Fish Oils in Vegetable Oils.—Heat some of the sample in a small covered crucible to about 130° C., and observe the odour given off. If much fish oil be present a characteristic fishy odour will be perceived. Measure out 50 cc. of the sample into a large test tube, and pass a current of dry chlorine gas through it, when a more or less reddish-brown colour will be produced if fish oils be present. Saponify 5 grms. of the sample as directed on page 251, but using absolute alcohol instead of the methylated (which gives a brownish colour with potash, owing to the methyl alcohol it contains), and dilute the saponified oil to about 50 cc. with water in a test tube, when, if fish oils be present, a more or less brownish colour is produced.

Valenta's Acetic Acid Test.—In this test the oil is brought into solution by boiling in 17 E acetic acid, and the temperature at which a turbidity is produced is observed. This being different for various oils, the test may be useful as confirming the presence of a particular oil which has been previously identified by other means. The test may be applied in the following manner:—5 cc. of the sample and 5 cc. of glacial (17 E) acetic acid are poured into a test tube, and the mixture heated to boiling, until all the oil has gone into solution; a thermometer is then immersed in the liquid, which is allowed to cool gradually, and the temperature at which a turbidity is produced is observed. The following results were obtained by Valenta, for various oils, by this test:—

Kind of Oil.	Temperature of Turbidity ° C.
Green Olive oil	85
Yellow ,,	111
Almond oil	110
Arachis ,,	112
Apricot Kernel oil	114
Sesamé oil	107
Melonseed oil	108
Cottonseed oil	110
Cod-liver ,,	101
Rapeseed oil	Not completely dissolved at the boiling point of acetic acid.
Mustardseed oil	
Wild Radishseed oil	
Palm oil	23
Laurel ,,	26 to 27
Nutmeg Butter	27
Cocoa Nut oil	40
Palmnut oil	48
Bassia oil	64·5
Beef Tallow	95
Pressed Tallow	114
Cacao Butter	105
Olive Kernel oil	Soluble at the ordinary temperature.
Castor oil	

Millian's Test for Detecting Cottonseed Oil.—Cottonseed may be detected in other oils by this test, and 5 per cent. of the oil mixed with rape or olive can be detected with certainty. The process consists in dissolving 5 cc. of the total fatty acids contained in a test tube in 2 cc. of absolute alcohol, heating to boiling in a water-bath, 2 cc. of a 2 E solution of silver nitrate are then added. If acids from cottonseed oil be present an immediate reduction of the nitrate occurs; the test tube may be kept near the boiling point of the alcohol for a few minutes to obtain the full development of the colour or precipitate. It is advisable in trying this test to heat 5 cc. of the same fatty acids with alcohol without the addition of the nitrate, so as to serve as a blank to be compared with the other.

Iodine Absorption Test for Oils.—This process of testing oils, which was first brought out by Baron Hübl[*] depends upon the fact that different oils, under the same conditions combine with varying quantities of iodine, owing to each oil possessing the glycerine ethers of the members of the three groups of fatty acids—viz., the *acetic*, *acrylic*, and *tetrolic* series, in different proportions; the acetic series being comparatively indifferent to the action of iodine, while the fats of the acrylic and tetrolic series readily unite with fixed quantities of the halogen. The following are details of the process:—

Weigh out into a 30 cc. beaker from 0·2 to 0·3 grm. of a drying oil, 0·3 to 0·4 grm. of a non-drying oil, or from 0·8 to 1·0 grm. of a solid fat. Dissolve in 10 cc. of chloroform, and pour into a 250 cc. stoppered flask. 20 cc. of a *standard* solution of iodo-mercuric chloride (prepared by dissolving 25 grms. of iodine and 30 grms. of mercuric chloride each in 500 cc. of 95 per cent. alcohol, and mixing the two solutions) are now added and the mixture well agitated. If the solution is not quite clear add a little more chloroform. If the mixture becomes nearly colourless after standing fifteen minutes add 10 cc. more of the iodine solution, well mix, and allow to stand for two hours. Now add 10 cc. of an aqueous solution of E potassic iodide, mix, dilute to about 200 cc., and titrate the free iodine still present with an $\frac{E}{10}$ standard solution of hyposulphite of soda (thiosulphate, $Na_2S_2O_3$), using 2 cc. of starch solution (1 in 100) as indicator of the end of the reaction.

A blank experiment must be made, using the same quantities of chloroform, iodine solution, &c., in order to obtain the true value of the hyposulphite solution, after which the amount of iodine absorbed by the sample is easily obtained, which should be expressed centesimally.

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[*] *Ding. Polyt. Jour.*, cclii., 281.

The following table shows the iodine absorption for various oils in percentages (Hübl and Möller):—

	Iodine degree.
Shark-liver oil	268·2
Manhaden ,,	170·8
Porpoise ,,	131·2
Seal ,,	103·4
Linseed ,,	158
Walnut ,,	143
Poppyseed ,,	136
Cottonseed ,,	106
Rapeseed ,,	100
Almond ,,	98·4
Castor oil	84·4
Olive ,,	82·8
,, ,,	81·3
Lard ,,	59
Palm ,,	51·5
Tallow ,,	40
Cocoa-nut oil	8·9
Cocoa-butter oil	34·4
Muskat-butter ,,	31·6
Mutton-fat oil	57·3
Lard ,,	55
Beef-suet ,,	38·4
Oleic acid ,,	86·2
Olein	82·3
Commercial stearin	1·7
Beeswax	5·3
Japanese wax	5·61
Bayberry tallow	1·38
Butter	31
Oleomargarine	55·3

Hübl obtained an assimilation of from 89·8 to 90·5 per cent. of iodine by chemically pure oleic acid, while the theoretical proportion required by the reaction $C_{18}H_{34}O_2 + I_2 = C_{18}H_{34}I_2O_2$ is 90·07 per cent.

The following are his conclusions deduced from the experiments*:—

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* *See* "Journal Soc. Chem. Ind.," 1884, p. 642.

(1) Linseed oil is distinguished by its high iodine, and any addition of a foreign oil must reduce that degree. By boiling linseed oil, its iodine degree is lowered, but the fusing point of its fatty acids is increased.

(2) An addition of 5 to 10 per cent. of cottonseed or rapeseed oil would be readily detected in nut or poppy oil, but not much less than 20 per cent. of linseed would be detected.

(3) Rapeseed oil as regards the iodine absorption is affected by the method of extraction and refining. The refined oil usually has an iodine degree 2 or 3 degrees below that of the raw product. A falsification with 15 per cent. of linseed oil would be detected with certainty.

(4) Castor oil possesses a very constant iodine degree—from 84·0 to 84·7, and is distinguished from all other oils and fats by this figure, by the points of fusion and solidification of its fatty acids (13° and 3° C. respectively), by the saponification test and by its ready solubility in alcohol and acetic acid.

(5) In twenty samples of olive oil, collected from various sources, the absorption of iodine varied only within 3 degrees. The addition of about 5 per cent. of a drying oil, or 15 per cent. of cottonseed, sesamé, arachis, or rape-seed, may be established with certainty.

(6) Animal fats, as is well known, are liable to alteration in respect to their consistency and proportion of oleic acid, owing to variety, age, and food of the animal whence derived. In the case of butter-fat the absorption of iodine varies between the limits of 26 and 35.

(7) When the nature of two fats in a mixture is known their proportion may be determined approximately by the formula :—

$$x = \frac{100\,(I - n)}{m - n}$$

Where x = the percentage of one fat.
y = ,, ,, the other.
I = iodine degree of the mixture.
m = ,, ,, ,, fat x.
n = ,, ,, ,, ,, y.

(8) The age of a fat, so long as great alterations have not taken place, does not affect its iodine absorption. If, how-

ever, an oil has become rancid and contains free acid, the iodine degree suffers considerable depression.

Saponification Equivalent of Oils.—It has been found that the various fatty oils require different amounts of potash for complete saponification, although, however, the difference in some cases is very slight, yet the number obtained is a careful criterion, coupled with the results of other tests, in judging the genuineness of a sample of oil. The "saponification equivalent" of an oil is the number of grams of the oil decomposed by one litre of a normal solution of an alkali. Two grams of the oil are weighed out into a bottle and saponified with 25 cc. of Std $\frac{E}{2}$ alcoholic potash solution, as described on page 251; the solution is then poured into a 100 cc. porcelain dish and the bottle washed out with a small quantity of alcohol, a few drops of phenolphthalein solution are then added, and the excess of KHO titrated with standard $\frac{E}{2}$ HCl. In all cases a blank titration is made with the alcoholic potash solution to ascertain its value, after which the amount of KHO taken up by the oil is easily calculated. The following table shows the saponification equivalent and percentage of KHO absorbed by various oils:—

	Saponification equivalent.	Percentage of KHO for Saponification.
Lard oil	285—296	19·1 to 19·6
Tallow oil	285—296	19·1 to 19·6
Neats-foot oil	285—300	19·2 to 20·3
Sperm oil	300—454	12·34 to 14·74
Whale oil	250—303	18·85 to 22·44
Menhaden oil	250—303	19·2
Cod-liver oil	250—303	18·5 to 21·3
Crude cottonseed oil	285—296	19·10 to 19·66
Rape oil	313—330	17·02 to 17·64
Castor oil	309—319	17·60 to 18·15
Olive oil	285—296	19·1 to 19·6
Rosin oil	290—330	17· to 19·3
Earth-nut oil	285—296	19·13 to 19·66

Hehner's Bromine Thermal Test for Oils.[*]—This is a more rapid method than the Hübl-iodine process, and has the advantage of approximately corresponding in thermal values for various oils—when multiplied by a constant—to the iodine values. The reagents required are methylated chloroform and liquid bromine, and the test is best carried out in a Dewar's jacketed vacuum tube, which can be arranged to tilt obliquely as in Fig. 67.

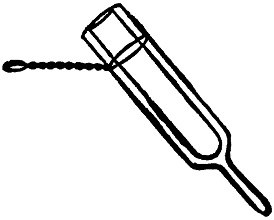

FIG. 67.

The sample, bromine, and chloroform are first brought to the same temperature, ascertained by a delicate thermometer graduated in ⅒ of a degree; one gram of the oil is then weighed out into the tube, and 10 c.c. of the chloroform added so as to dissolve the oil. When it is assured, the temperatures of the reagents are equalized; 1 c.c. of the bromine is withdrawn from the bottle by means of a pipette, having a little soda lime-tube fitted to the upper end, and attached to a piece of rubber tube for a mouth-piece, and added to the solution of the oil, and immediately stirred with the thermometer until no further rise in temperature takes place; the increase in the temperature is the bromine thermal value.

The following are some of the figures obtained, the calculated iodine number being the rise in temperature multiplied by the constant 5·5: —

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[*] *Vide* Hehner, *Analyst*, 1895, p. 146, and Archbutt, *Jour. Soc. Chem. Indus.*, April, 1897.

	Oil or Fat.	Rise of Temperature with Bromine.	Hübl figure.	Calculated Iodine number.
1.	Lard	10·6	57·15	58·3
2.	,,	10·4	57·13	57·2
3.	,,	11·2	63·11	61·6
4.	,,	11·2	61·49	61·6
5.	,,	11·8	64·69	64·9
6.	,,	11·8	63·96	64·9
7.	,,	10·2	57·15	56·1
8.	,,	10·4	57·80	57·2
9.	,,	9·0	50·38	49·5
10.	,,	11·0	58·84	60·5
11.	,, + 10 °/₀ cotton oil	11·6	64·13	63·8
12.	,, fatty acids	10·4	59·6	57·2
13.	,, ,, ,,	11·0	59·15	60·5
14.	Mutton fat (kidney)	8·1	44·48	44·5
15.	,, ,, (flare)	7·6	39·7	41·8
16.	Butter	6·6	37·07	36·3
17.	,,	7·0	38·60	38·5
18.	,, fatty acids	6·2	36·5	34·1
19.	Almond oil	17·6	96·64	96·68
20.	Olive oil	15·0	80·76	82·5
21.	Maize oil	21·5	122·0	118·2
22.	Cotton oil	19·4	107·13	106·7
23.	Castor oil	15·0	83·77	82·5
24.	Linseed oil	30·4	160·7	167·2
25.	,, ,,	31·3	154·9	172·0
26.	Rape oil	18·4	88·33	101·2
27.	,, ,,	17·6	77·20	96·8
28.	Cod-liver oil	28·0	144·03	140
29.	Oil sent as olive oil	19·0	108·5	104·5
30.	,, ,, ,,	19·2	105·7	105·6
31.	,, ,, ,,	18·9	105·7	103·9

PART VI.

MATERIALS USED IN THE MANUFACTURE OF RAILWAY GREASE.

Railway grease may be made from tallow, palm oil, petroleum residuum, caustic or carbonated alkali, and soap. A known weight of alkali (which is determined according to the percentage of free fatty acids present in the tallow and palm oil), is first dissolved in an appropriate quantity of water contained in an open boiler, into which is fitted a perforated coil of iron tubing, through which steam can be made to issue. A weighed quantity of palm oil is next added, and thoroughly stirred until the free fatty acids are completely neutralized, which can be known by the abatement of effervescence, due to CO_2 from the decomposition of the alkali; a definite weight of tallow is next added, and the mixture "boiled" up with steam for some time, when the free fatty acids of the tallow thus become neutralized. After the addition of soap, and steaming, the grease is run off through sieves into vats, or, if a black grease is required, about 40 per cent. of Russian petroleum residuum is previously added.

The quality of the materials used in the manufacture of grease is of great importance in ensuring uniformity of working, and in obtaining a homogeneous product. A source of trouble in practical working is the great difference in the percentages of free fatty acids in different consignments of palm oil, Lagos brand containing about 12 per cent. of free fatty acids, expressed as palmitic acid, while the brand known as Salt Pond may contain as much as 80 per cent. of free fatty acids.

The following may be taken as being the average composition of good yellow and black railway grease:—

	Yellow. Per cent.	Black. Per cent.
Water	37	25
Tallow	30	17
Palm oil	10·5	4
Soap (containing 30 °/₀ water)	21	13·5
Petroleum residuum	—	40·0
Soda ash	1·5	1·5
	100·0	100·0

Grease used on the German railways has the following composition:—

	Per cent.
Tallow	24·6
Palm oil	9·8
Rape-seed oil	1·1
Soda	5·2
Water	59·3
	100·0

Austrian Grease:—

	Tallow.	Olive Oil.	Old Grease.
For winter	100	20	13
For spring and autumn	100	10	10
For summer	100	1	10

Tallow.

Australian mutton tallow and Russian tallow are much used in the manufacture of grease, and in judging its quality it becomes necessary to know the amount of moisture, free fatty acids, suspended matter (mineral and organic), the total fatty acids, together with the determination of the melting and congealing points of the sample, and also of the total fatty acids.

Estimation of Moisture.—Weigh into a tared beaker, supplied with a glass rod, 10 grms. of the fairly averaged sample, dry in an oil bath or air oven for one hour, at a temperature of 110° C., allow to cool and reweigh; replace in the oil bath or air oven for another hour, cool, and again weigh; if there is no considerable difference between this and the last weighing the loss is taken as moisture. The normal amount of moisture in tallow ranges from 0·5 to 2 per cent.

Estimation of Suspended Matter.—If the amount of suspended matter is at all considerable—which could be judged when the tallow is melted—the tallow left from the estimation of moisture is melted, and passed through a tared ashless filter-paper contained in a hot water funnel, after which it is transferred to an ordinary filter stand, and the remaining portion of tallow and suspended matter in the beaker treated with methylated ether, and filtered, the filter being washed free from fat with ether, dried and weighed; after which the filter and its contents can be ignited in a weighed crucible, and the residual mineral matter weighed. The total suspended matter in a tallow should not exceed 0·3 per cent.

Estimation of Free Fatty Acids.—50 grms. of the sample are weighed into a 180 cc. flask, and 100 cc. of pure methylated alcohol (to which are previously added three drops of an alcoholic solution of phenolphthalein and two drops of E NaHO solution, which should give a permanent pink coloration) are added; the mixture is then placed on top of a water oven, and when the tallow is seen to be melted, it is vigorously stirred so as to dissolve all the free fatty acids. A standard solution of E sodic hydrate, contained in a burette, is now gradually run into the solution until a pink coloration (which remains permanent on stirring for five minutes) is produced. Note the volume required. 1 cc. of E NaHO = 0·282 grm. free oleic acid; cc.'s required × ·282 × 2 = percentage of free oleic acid. The usual range of free fatty acids in tallow is from 1 to 5 per cent., and in samples which have been kept for some years, and which are characterised with a very offensive rancid odour, may contain as much as 20 per cent. Sometimes, however, the free fatty acids are abnormally high, due to adulteration with cotton-seed oil, stearic acid, and the acids from wool grease.

Estimation of Total Fatty Acids.—5 grms. of the sample are saponified as directed on page 251, and the total fatty acids determined as described.

The Melting Point and Congealing Point of the Sample, and of the Total Fatty Acids therefrom.—These are determined by the method described on page 254. The melting point of Australian tallow is from about 45° to 50° C., and the congealing point from 40° to 47° C. The melting point of the total fatty acids is often 49° C., and the congealing point 47° C.

Calculation of amount of Soda-ash required to neutralize the Free Fatty Acids in 1 cwt. of Tallow.—We shall suppose the soda-ash to contain 47·7 per cent. of combined Na_2O and the tallow to contain 4·8 per cent. free oleic acid. Now two molecules of oleic acid require one molecule of sodic oxide, since

$$\underbrace{2\,C_{18}H_{34}O_2}_{564\text{ reqs.}} + \underbrace{Na_2O}_{62} = 2\,NaC_{18}H_{33}O_2 + H_2O.$$

Now 1 cwt of the tallow contains ·048 cwt. $C_{18}H_{34}O_2$; then 564 : ·048 : : 62 : x— = ·00528 cwt. pure Na_2O required, but the soda-ash only contains 47·7 per cent. pure Na_2O; then, 47·7 : 100 : : ·00528 : x = ·01107 cwt. or ·01107 × 112 = 1·24 lbs. soda-ash required to neutralize the free fatty acids in 1 cwt. of the tallow.

Palm Oil.

Palm oil is obtained from the fruit of the *Avoira elais* or *Elais Guineenis*, and is imported largely into this country from the West Coast of Africa for the purpose of making grease, soaps and candles, &c. Its specific gravity is ·920 to ·927 at 15° C. and about ·856 at 100° C., and it melts at about 40° C., the total fatty acids melting at from 41 to 46° C.

The colour of palm oil varies from orange yellow to brownish yellow. In consistency it varies from that of vaseline to hard tallow, owing to the variation in the proportion of free fatty acids. Palm oil may be assayed in a similar manner to tallow, the free fatty acids being expressed as palmitic acid, ($C_{16}H_{32}O_2$) 5 grms. instead of 50 being taken. For railway grease the amount of free fatty acids present is not a serious item, since they are neutralized in the process of manufacture. The oil in its raw state, however, quickly corrodes iron, steel, &c. The following is a table by Archbutt giving the proportions of free fatty acids in various brands of palm oil.

	Free Fatty Acids, per cent. Expressed as Palmitic.
Salt Pond	78·9
Unknown	72·0
Refined	55·8
Brass	53·2
New Calabar	52·2
Fernando Po	40·5
Half Jack	35·7
Half Jack	24·4
Bonny	21·5
Lagos	11·9

The following is what the author considers an ideal palm oil for grease making:—

Free fatty acids	30 per cent.
Total „ „	95 „
Water, below	0·5 „
Suspended matter, below	·3 „
Melting point of sample	41° C.
Congealing point of sample	36° C.
Melting point of total fatty acids	42° to 47° C.
Congealing „ „	43° to 41° C.

The Lagos brand contains the least proportion of free fatty acids, but it is likewise the most expensive, and therefore it is more economic to use for grease making a palm-oil containing a fair proportion of free fatty acid at a reasonable price. It could, of course, be arranged that there should always be the same proportion of neutral palm fat present in the grease, by altering the proportion of added soap according to the amount of soap formed by the neutralization of the free fatty acids. There should be no difficulty in the making of grease with palm oil so long as the proportion of the free fatty acids keeps below 40 per cent.

Petroleum Residuum.

The best petroleum residuum applicable to grease-making is that imported from Russia. If the distillation has not been carried too far, it is of a fine glossy silky nature ; and when rubbed on the hand, it exhibits a good greasy consistency. On the other hand, if the distillation has been carried on to the extent that the last portion of lubricating oil has been removed, a rough-feeling product is obtained which is inferior for the purpose.

Petroleum residuum is now much used in the manufacture of railway grease, and it appears to be a very economic introduction. It can be got for something like £6 per ton ; and moreover, the grease made with it lasts longer, and has less action on the bearings, journals, &c., than the ordinary yellow grease. The most important point in buying residuum is to get it of the required viscosity ; many of the residuums in the market become very thin when the temperature rises a litttle, and grease made with them is of little value, since on attaining the temperature of the bearings it separates and runs away in waste. The viscosity of residuums for grease-making should be determined at 180° C., and the best for the purpose takes about 546 seconds for 50 cc. to run out of Redwood's viscosimeter. The amount of suspended matter if large must be determined, and also the loss on exposing about 1 grm. of the sample contained in a watch glass of 2½ inches diameter, in a water oven for twelve hours, the loss being due to water and any light oil that has volatilized, an amount which rarely exceeds 2 per cent. Sometimes residuums contain a considerable amount of water ; in such cases it is best to treat a known weight with petroleum ether in a graduated measure, and note the volume of water that has separated out.

Soap.

Palm soap containing 28 per cent. of water and 8 to 13 per cent. of resin as soap is much used for grease-making, but it is questionable whether a much cheaper soap could not be used with equal advantage, containing no resin, which is of little use as a lubricant.

METHOD OF ANALYSIS.

Estimation of Water.—The amount of water in soaps considerably varies, some hard soaps containing 15 per cent., and inferior varieties as much as 80 per cent. Weigh out 2 grms. of the fairly averaged sample into a tared beaker supplied with a pointed glass rod, and dry for one hour at 50° C.; and finally gradually increase the temperature to 110° C., with frequent stirring until all water has been driven off; about three hours in all being in most cases sufficient to completely expel water. The loss = water.

Estimation of Total Combined Fatty and Resin Acids.—5 grms. of the average sample are dissolved in 100 cc. of hot water and poured into a separator, 20 cc. of *standard* E HNO_3 are then added, and the mixture well shaken up; the soap is thus decomposed, fatty acids and any free unsaponified fat being liberated, the soda or potash combining with the nitric acid forming nitrate of soda or potash. 50 cc. of methylated ether are now added, well shaken, and allowed to settle. The aqueous solution is tapped off into another separator, 20 cc. more ether added to it and separated as before; the two ethereal solutions are mixed and washed with water, and separated, adding the aqueous solution to the main portion. Reserve aqueous solution. The ethereal solution containing the fatty acids, &c., is poured into a weighed flask and attached to a Liebig's condenser, the ether distilled off, and the residue carefully dried and weighed; any unsaponified fat and resin as found in another portion, must be subtracted from this weight. Of course the fatty acids as thus found do not exist in this form in the soap, but as fatty anhydrides; to obtain the corresponding quantity of fatty anhydrides, multiply the weight of fatty acids found by ·966.

Estimation of Total Soda (*Na_2O*.)—To obtain the total amount of soda present, free and combined, the reserved aqueous solution from the above determination is taken: the excess of E nitric acid therein is titrated back with a standard E solution of NaHO, using litmus paper as an indicator.

Each cc. of E HNO_3 neutralized = ·032 grm. Na_2O.

The solution could now be divided into two halves, and if necessary the chlorides could be estimated in one by precipitation with nitrate of silver, and sulphates in the other by precipitation with barium chloride in the usual manner.

Estimation of Resin.—The best method for the determination of resin is that devised by Gladding, depending upon the solubility of silver resinate in an alcohol-ether solution, and the comparative insolubility of silver oleate, stearate, palmitate, &c., in the same solution. Weigh out 0·5 grm. of the total fatty and resin acids, as obtained in their determination, into a small flask, dissolve in 20 cc. of absolute alcohol, add two or three drops of phenolphthalein, then add drop by drop a concentrated alcoholic solution of potash till a pink coloration is produced, and then add two drops more, and keep near the boiling point for ten minutes to ensure complete saponification of any neutral fat; pour out into a graduated 100 cc. Muter tube, Fig. 68, dilute to 100 cc. with anhydrous ether, and thoroughly mix. Now add 0·5 grm. of pure silver nitrate in the state of the finest powder, and thoroughly shake for about ten minutes to ensure complete reaction. Allow the precipitated silver soaps to subside. About 70 cc. or any known quantity of clear ethereal solution are tapped off into a separator, and shaken up with about

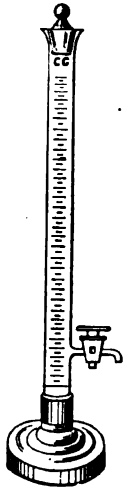

FIG. 68.

0·1 grm. more powdered silver nitrate to ensure complete formation of silver soaps. If any flocculent precipitate is produced the solution is again run into the Muter tube and shaken up with 0·3 grm. more of the powdered silver nitrate, allowed to settle, and a known volume tapped off into a separator as before. 20 cc. of 4 E HCl are now added to the liquid, and the mixture vigorously shaken up. The soluble silver soaps are thus decomposed, silver chloride is precipitated and collects at the bottom of the aqueous solution, while the resin acids and small quantities of oleic acid, &c., are dissolved in the ethereal layer. When complete separation is ensured, the aqueous solution with the precipitate is tapped off, the ethereal solution distilled off in a weighed flask, and the residue of resin, &c., dried at 100° C. and accurately weighed. Now, since the silver soaps of oleic, stearic acids, &c., are to an appreciable extent soluble in the ethereal liquor, a correction must be made for the solubility; the amount dissolved varies with the nature of the fatty acids.

The following table showing the average variation of solubility of different fatty acids as silver soaps, was obtained by Wright and Thompson :—

	Milligrams of Fatty Acids in 10 cc. of Solvent.
Pure oleic acid	1·20
Pure stearic acid	1·16
Mixed stearic and oleic acids	1·91
Acids from cottonseed oil	2·69
Acids from castor oil	5·39
Acids from cocoa-nut oil	1·80
Acids from linseed oil (Allen)	2·31

An idea of the nature of the fatty acids present having been obtained from their melting point, specific gravity, &c., the operator could judge which was the most correct figure to take for subtraction. The method gives good results with a little practice, but unless great care is exercised a very erroneous result may be obtained.

Estimation of Free Fat.—Owing to incomplete saponification of the fat used in the manufacture of soap, there is sometimes fat present in the free state. To determine this, the dry residue left from the estimation of water is detached by means of a flexible steel spatula, powdered, brushed into a 50 cc. wide mouth stoppered bottle, 30 cc. of methylated ether added, and the stopper tied down. It is now heated to about 40° C., well shaken for about a quarter of an hour, and allowed to cool; the stopper is then withdrawn, the clear ethereal solution decanted through a filter into a separator, the residue exhausted twice more with 30 cc. of ether, and filtered as before. The ethereal solution containing any free fat, free fatty acids, a little soap, &c., is now washed three times with 20 cc. of distilled water, the purified ethereal extract distilled, and the residuum of free fat, &c., weighed and tested for mineral oils and waxes, &c.

The following is a tabular scheme given by Dr. Leeds * for the complete analysis of soaps:—

* *Chem. News*, xlviii., 67.

<table>
<tr><td colspan="11">Weigh out 5 grms. of sample. Dry at 110° C. Loss = water. Treat with petroleum ether.</td></tr>
<tr><td rowspan="5">*Extract* is uncombined fat. Dry at 110° C., and weigh.</td><td colspan="10">Residue is soap and mineral constituents. Treat with alcohol.</td></tr>
<tr><td colspan="5">*Extract* is soap (fatty anhydride, resin, and combined alkali), glycerine, and free alkali. Add two or three drops of phenolphthalein, and, if necessary, titrate with $\frac{E}{10}$ H_2SO_4.</td><td colspan="5">*Residue.*—Na_2CO_3, NaCl, Na_2SO_4, sodium silicate, starch and insoluble residue. Wash with 60 cc. water.</td></tr>
<tr><td rowspan="3">$\frac{E}{10}$ H_2SO_4 used corresponds to free caustic alkali. Calculate as NaHO.</td><td colspan="4">Add a large excess of water and boil off the alcohol. Decompose with an excess of a known volume of standard E H_2SO_4. Boil, filter, and wash.</td><td colspan="4">*Filtrate.*—Na_2CO_3, NaCl, Na_2SO_4, and sodium silicate. Divide into four equal parts.</td><td rowspan="2">*Residue.*—Starch and insoluble residue. Dry the filter and weigh; the weight is the starch and insoluble residue starch. Convert into $C_6H_{12}O_6$, titrate with Fehling's solution, subtract the weight of starch found, and the difference is the insoluble mineral constituents.</td></tr>
<tr><td colspan="2">*Filtrate.* Combined soda and glycerine. Titrate excess of acid back with standard E NaHO.</td><td colspan="2">*Residue.* Fatty acids and resin. Dry at 110° C., and weigh. Dissolve aliquot part in 20 cc. alcohol, and saponify with slight excess of NaHO, using phenolphthalein as indicator, boil, cool, add ether to 100 cc. in a Muter tube. Decompose with ·5 grm. powdered $AgNO_3$, and shake well; allow to settle.</td><td>Na_2CO_3. Titrate with standard $\frac{E}{10}$ H_2SO_4, and calculate as Na_2CO_3.</td><td>NaCl. Titrate with standard $AgNO_3$, or weigh as AgCl. Calculate as NaCl.</td><td>Na_2SO_4. Precipitate with $BaCl_2$, weigh the $BaSO_4$, and calculate for Na_2SO_4.</td><td>Sodium silicate. Decompose with HCl, and determine soda combined in silicate and silica.</td></tr>
<tr><td>E H_2SO_4 used corresponds to the combined Na_2O in soap.</td><td>After titration evaporate to dryness on the water bath. Treat with absolute alcohol, evaporate the alcoholic solution in a tared flask, and weigh any glycerine.</td><td>*Precipitate* is oleate, palmitate and stearate of silver.</td><td>*Solution.* Resinate of Ag. Filter 50 cc. from total 100 cc., decompose with 20 cc. of 4 E HCl. Allow to settle. Evaporate an aliquot part in tared beaker, dry at 110° C., and weigh. After applying the correction the weight = resin; subtract this from fatty acids + resin present; remainder = fatty acids.</td><td colspan="5">SYNOPSIS OF RESULTS.
Per cent.
• Fatty anhydrides . . . . . .
Resin acids . . . . . .
Soda, existing as soap . . . . .
Water . . . .
Sodic carbonate . . . . . .
„ hydrate . . . . . .
„ chloride . . . . . .
„ sulphate . . . . . .
Silica . . . .
Silica as silicate . . . . . .
Lime, &c. . . . .
• Equals per cent. fatty acids.</td></tr>
</table>

TABLE OF ANALYSIS OF VARIOUS SOAPS. (HOPE).

Description of Soap.	Origin.	Fatty and Resin Anhydrides.	Soda Na_2O existing as Soap.	Silica.	Soda as Silicate.	Caustic and Carbonate of Soda.	Sodic Chloride.	Sodic Sulphate.	Lime, Oxide of Iron, &c.	Water.	Total.	Fatty and Resin Acids.
White, No. 1 .	Tallow	69·06	8·98	0·01	nil	0·27	0·49	0·16	0·07	21·14	100·18	71·20
,, ,, 2 .	Tallow and Cocoa-nut Oil	60·50	6·82	0·06	,,	0·06	0·11	0·12	0·16	32·20	100·03	62·36
,, ,, 3 .	Do.	55·71	6·90	0·03	,,	0·92	0·18	trace	0·08	36·54	100·36	57·44
,, ,, 4 .	Do.	44·27	6·23	7·02	2·36	0·75	0·32	0·34	0·34	38·14	99·77	45·64
Cold Water, No. 1	Tallow, Resin, and Cottonseed Oil	71·30	7·98	1·07	0·48	0·75	0·36	0·30	0·16	17·44	99·84	73·50
,, ,, ,, 2	Do.	49·95	7·00	2·34	1·01	0·33	0·51	nil	0·50	38·18	99·82	51·50
Olive Oil, No. 1 .	Olive Oil	71·20	7·58	0·06	0·03	0·22	0·66	0·17	0·20	19·70	99·82	73·40
Marseilles, No. 1 .	Chiefly Olive Oil	62·66	7·27	0·06	0·03	0·77	0·76	0·30	0·16	28·20	100·21	64·60
Palm Oil, No. 1 .	Palm Oil	59·28	6·65	0·42	0·01	0·39	0·47	0·13	0·16	32·35	99·86	61·08
Mottled . .	Palmnut Oil	38·89	5·76	6·40	1·29	1·62	1·78	0·72	0·03	38·70	95·19	40·10
Satinet . . .	Tallow and Resin	59·92	6·76	0·02	nil	0·92	0·41	0·37	0·05	31·30	99·75	61·77
Glasgow Almond .	Do.	42·41	4·14	5·64	1·59	2·76	0·37	trace	0·14	42·88	99·93	43·72
Pale Resin, No. 1	Do.	60·90	7·22	0·04	nil	0·10	0·46	0·12	0·02	31·22	100·08	62·78
,, ,, ,, 2	Do.	48·20	5·00	0·42	0·18	0·15	0·65	0·10	0·10	45·00	99·80	49·65
,, ,, ,, 3	Do.	39·92	4·70	0·62	0·25	0·20	1·48	0·18	0·15	52·40	99·90	41·15
Milling . .	—	63·06	7·25	0·02	nil	0·10	1·65	0·15	0·30	27·47	100·00	64·95
Yellow (for foreign markets)	—	10·90	1·36	0·03	,,	trace	2·57	0·56	0·14	84·00	99·56	11·20
Marine (for emigrants)	Palmnut Oil	19·42	3·11	9·00	3·98	3·00	5·13	0·35	0·16	53·32	97·47	20·02

Assay of Caustic Soda (NaHO).

Weigh out quickly 10 grms. of the fairly averaged sample, dissolve in the purest distilled water free from CO_2, dilute to 1,000 cc. at 15° C. in a graduated stoppered flask, thoroughly mix, and allow any sediment to subside.

Estimation of Total Alkali.—100 cc. (= 1 grm. sample) of the above clear solution are measured off and poured into a 200 cc. beaker; 28 cc. of *standard* E H_2SO_4 are now run in from a burette, and the mixture boiled for five minutes with the cover on. It is then cooled, a few drops of litmus solution added, and *standard* E NaHO run in gradually from a burette, stirring until the last drop added just turns the red solution blue. Note the volume required, subtract this amount from 28, and the quantity of E acid neutralized by 1 grm. of sample is obtained.

Each cc. neutralized = ·031 grm. soda (Na_2O).

Example.—A sample of caustic soda treated as above required 8·8 cc. of E NaHO to neutralize excess of acid, then:—

28 – 8·8 = 19·2 cc. E H_2SO_4 neutralized.
19·2 × 0·031 = ·5952 grm. Na_2O in 1 grm.
·5952 × 100 = 59·52 per cent. Total Na_2O.

Estimation of Na_2O present as Hydrate and Carbonate.—Take 250 cc. of the original solution (= 2·5 grms. of sample), pour into a graduated 500 cc. flask, add 40 cc. of E $BaCl_2$, dilute to 500 cc. at 15° C., stopper, thoroughly mix, and allow any precipitate of carbonate of barium to subside; decant off 250 cc. (= 1·25 grm. original sample) of the clear solution into a 400 cc. beaker, add three drops of phenolphthalein solution, and run in *standard* E HCl from a burette until the pink colour has just disappeared. Note the amount required.

Example.—250 cc. of the original solution were treated as above, and 22·6 cc. of standard E HCl were required to decolourize the solution.

1 cc. of E HCl = ·031 grm. Na_2O.
22·6 × ·031 = ·7006.
$\frac{·7006}{1·25}$ = ·5605 grm. Na_2O as NaHO in 1 grm. sample.
·5605 × 100 = 56·1 per cent. Na_2O as NaHO.

The total Na_2O was 59·52 per cent.

Then—

59·52 − 56·1 = 3·42 per cent. Na_2O as carbonate.

57 to 60 per cent. of total soda, and 2 to 3 per cent. soda as carbonate are frequent proportions in the commercial article.

Assay of Soda Ash (Na_2CO_3).

Weigh out 10 grms. of sample, dissolve in 300 cc. of distilled water, filter into a graduated 500 cc. stoppered flask, wash the residue with distilled water until the washings are no longer alkaline, and dilute to 500 cc. at 15° C. The solution is then thoroughly mixed. 100 cc. (= 2 grms. sample) are accurately measured out and poured into a 200 cc. beaker, and 37 cc. of *standard* E H_2SO_4 gradually added from a burette; the solution is now boiled for five minutes with cover on to eliminate CO_2, allowed to cool, a few drops of litmus solution added, and the free acid titrated by a standard E solution of sodic hydrate until the last drop produces a blue coloration. Note the volume required.

Example.—10 grms. of a sample of soda ash were treated as above; on titrating the free acid with standard E NaHO 6·0 cc. were required.

Then—

37 − 6 = 31 cc. of standard E H_2SO_4 neutralized.
1 cc. of E H_2SO_4 = ·031 grm. Na_2O.
31 × ·031 = 0·961 grm. Na_2O in 2 grms. sample.
$\frac{0·961 \times 100}{2}$ = 48·05 per cent. combined Na_2O.

or,

$\frac{31·41 \times 48·05}{31}$ = 48·7 English degrees.

A great number of commercial soda ashes examined by the writer ranged from 46 per cent. Na_2O to 52 per cent. Na_2O.

The following is a *complete* analysis of Glasgow refined soda ash (*Brown*).

Sodium carbonates	80·92
,, hydrate	3·92
,, silicate	1·32
,, aluminate	1·01
,, sulphate	7·43
,, sulphite	1·11
,, thiosulphate	trace
,, sulphide	0·23
,, chloride	3·14
Insoluble matter	0·77
	99·85

Scheme of Analysis.—The following is a systematic scheme for the analysis of railway grease, &c., and also dirty grease, *i.e.*, grease that has done its duty as a lubricant and which contains metals, &c.

SCHEME OF ANALYSIS.

<table>
<tr><td colspan="3">Ten grms. of the sample are stirred with ether in a beaker till <i>thoroughly disintegrated</i>. The mixture is then rinsed into a separator and shaken repeatedly with quantities of 10 cc. each of E HCl till all metals are removed. (Iron and aluminium can only be removed by <i>strong</i> acid. Ether dissolves ferric chloride from its aqueous and <i>dilute</i> acid solution.) The ethereal solution which remains is then washed three or four times with water in quantities of 10 cc. each time.</td></tr>
<tr><td rowspan="3"><i>Acid Extract.</i>—Wash with ether to remove oil mechanically carried over, and add the ether to the main quantity. Run the acid solution into a basin and let the ether spontaneously evaporate; then take to dryness, redissolve, and estimate the <i>Metals</i> in the usual manner.</td><td colspan="2"><i>Ethereal Solution.</i>—Shake with about 50 cc. of "soda mixture," caustic soda 20 grms., methylated spirit 100 cc. and dilute to 1 litre with water.</td></tr>
<tr><td rowspan="2"><i>Alkaline Extract.</i>—Wash with ether and add the ether to the main quantity. Then acidify the solution, shake with ether, evaporate the ether and weigh the <i>Fatty Acids</i> (and the greater part of the <i>Resin</i>) which existed as soap in the grease, and perhaps partly in the free state. These can be examined further as desired.</td><td><i>Ethereal Solution.</i>—Evaporate, and weigh the residual oil as control. Then saponify, and shake the alkaline solution with ether.</td></tr>
<tr><td><table><tr><td><i>Ethereal Extract.</i>—Consists of <i>Hydrocarbon Oils</i>, <i>Cholesterin</i> and <i>Unsaponifiable Matters</i>, <i>Higher Alcohols</i>, <i>&c.</i></td><td><i>Alkaline Solution.</i>—Acidify, and estimate fatty acids existing in the grease as <i>Neutral Oil or Fat.</i> If the grease contained resin, part of it will be found here.</td></tr></table></td></tr>
</table>

PART VII.

GASWORKS PRODUCTS.

COAL GAS.

ENORMOUS quantities of coal are now consumed in the manufacture of gas for lighting, heating, and motive purposes. The manner in which it is at present produced is at once simple and economic, although at first considerable difficulties had to be overcome in obtaining a systematic process which would supply a continuous yield of gas, free from objectionable impurities such as sulphuretted hydrogen, carbon bisulphide, carbonic acid, air, &c. Fig. 69 is a sketch illustrating the principle upon which gas is produced in a modern gas-works.

The gas coal is introduced into the cylindrical fire-clay retorts *a a*, contained in a furnace in which they are heated to a bright red heat; the products of distillation, gas, tar, and ammoniacal liquor, pass up the ascension pipes attached to each retort, and through the hydraulic main B, and from thence descend through the pipe *d*, through the tar-pit *c*; here the tar and ammoniacal liquor collect, and the gas passes through the series of bent tubes *h h h*, where the gas is cooled and any tar, &c., still present in the gas, is condensed. Before reaching the holder G the gas has now to be purified from carbonic acid, and sulphuretted hydrogen, and these impurities are eliminated by allowing the gas to percolate through slaked lime contained on a series of shelves enclosed in iron cases; oxide of iron mixed with saw-dust is now also largely used for the purpose. Carbonic acid is a very undesirable constituent of coal

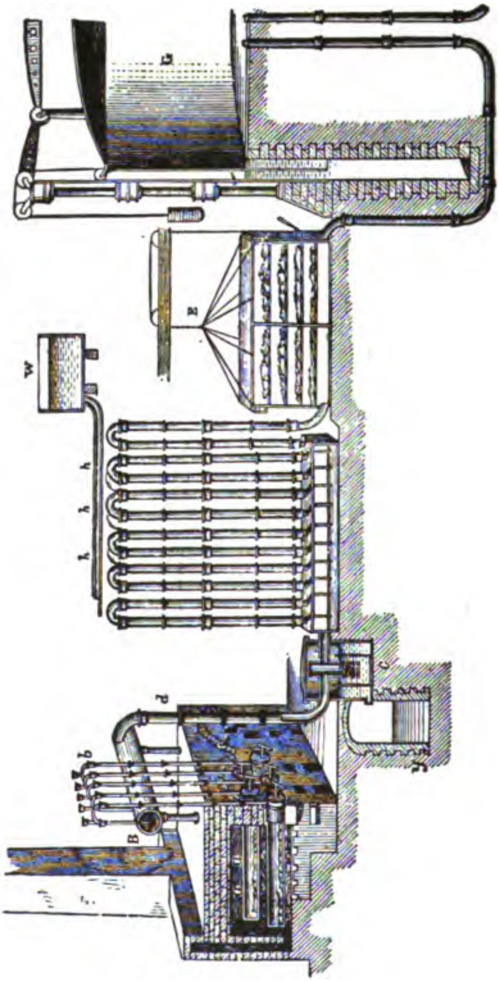

FIG. 69.

gas; if present only to the extent of one per cent. the illuminating power of the gas is diminished something like 6 per cent., while the presence of sulphuretted hydrogen and other gaseous sulphur compounds give rise to sulphurous acid, which rapidly corrodes oil paintings, metal work, &c.

The illuminating power of coal gas depends upon the nature and amounts of hydrocarbons present, which are regulated by (1) the quality of coal used, (2) the temperature of distillation, (3) the length of time the gas is in contact with the heated sides of the retorts and the liquid products of the distillation. The theory which is now generally accepted as the cause of luminosity of coal-gas flames is that first advanced by Sir Humphry Davy, so long ago as 1817, who stated that "the intensity of the light of flames depends principally upon the production and ignition of solid matter in combustion." In the case of coal gas, candles, or oil burning, the solid matter present is of course carbon, precipitated in the flame owing to a want of sufficient oxygen for its oxidation, here it is heated to incandescence by the burning of the hydrogen, marsh gas, &c., present in the gas. The higher the temperature the carbon particles are heated the greater will be the luminosity. Pressure plays an important part in the intensity of light emitted by flames. It has been found that by an increase in the pressure of the atmosphere in which a flame is burning there is an increase in the luminosity, so that the light emitted by a candle on the top of a mountain is less than that which is produced at the bottom, although the consumption of the candle may be the same. Dr. Frankland supposes the cause of the action of pressure upon the luminosity to be due to the alteration of density and alteration of the mobility of the oxygen molecules in the air. Wartha, however, attributes it to the effect of pressure on the disassociation point of the hydrocarbons burning in the flame, which takes place more rapidly with augmented pressure, and therefore increasing the rate of the liberation of carbon.

In obtaining the illuminating value of gas advantage is taken of the fact that the intensity of light varies as the square of the distance. The standard of light generally used is that of a sperm candle, six to the pound, burning 120 grains per hour, as compared with the light given by the gas burning at the rate of 5 cubic feet per hour. The apparatus used by the gas referees is the Bunsen photometer, as modified by Letheby, of which Fig. 70 is a sketch.

It consists of an experimental meter through which the quantity of gas burnt in a given time is registered. The gas is burnt at one end of a graduated bar, and a couple of candles suspended on one side of a balance are burnt at the other, and a disc of prepared paper contained within a sliding box is saddled upon the bar, and which can be made to move between the two lights, until the opposite sides are equally illuminated, the bar being graduated according to the law that the intensity of the luminosity varies as the square of the distances. Various precautions have to be taken in order to obtain an accurate result. The temperature and pressure must be observed, since they influence the result; the amount of sperm burnt must be ascertained; the volume of gas to be tested must be accurately measured, and regulated by means of a governor, so as to burn 5 cubic feet per hour. The testing must be performed in a dark room.

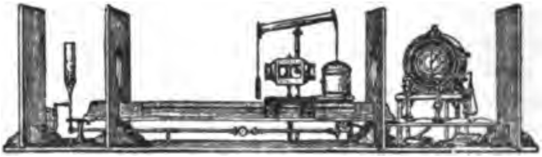

FIG. 70.

Statutory Regulations for Testing the Illuminating Power of Gas.—The provisions in Schedule A of the Gas Works Clauses Act, 1871, in regard to the apparatus for, and the mode of testing, the illuminating power of gas, are as follows:—

Regulations in Respect of Testing Apparatus.—The apparatus for testing the illuminating power of the gas shall consist of the improved form of Bunsen's photometer, known as Letheby's open 60-inch photometer, together with a proper meter, minute clock, governor, pressure gauge, and balance.

The burner to be used for testing the gas shall be such as shall be prescribed.

The candles used for testing the gas shall be sperm candles of six to the pound, and two candles shall be used together.

Mode of Testing for Illuminating Power.—The gas in the photometer is to be lighted at least fifteen minutes before the testings begin; and it is to be kept continuously burning from the beginning to the end of the tests.

Each testing shall include ten observations of the photometer, made at intervals of a minute.

The consumption of the gas is to be carefully adjusted to 5 cubic feet per hour.

The candles are to be lighted at least ten minutes before beginning each testing, so as to arrive at their normal rate of burning, which is shown when the wick is slightly bent, and the top glowing. The standard rate of consumption for the candles shall be 120 grains each per hour. Before and after making each set of ten observations of the photometer, the gas examiner shall weigh the candles, and if the combustion shall have been more or less per candle than 120 grains per hour, he shall make and record the calculations requisite to neutralize the effects of the difference.

The average of each set of ten observations is to be taken as representing the illuminating power of that testing.

Instructions of the London Gas Referees as to the Times and Mode of Testing for Illuminating Power.—The testings for illuminating power shall be three in number daily. The photometers to be used in the testing places shall be the improved forms of the Bunsen Photometer, prescribed and certified by the referees. The burner attached to the photometer shall be a standard burner, corresponding to that which has been deposited with the Warden of the Standards, in accordance with Section 37 of the Gaslight and Coke Company's Act, 1876. A description of the standard burners to be used for testing common gas and cannel gas respectively is given. (See page 311).

The gas in the photometer is to be kept burning for at least fifteen minutes before any testing is made.

The disc used in the photometer shall be either the Leeson or the Bunsen disc. The chimneys to be cleaned daily. The candles shall be such as are described in Section 25 of the Metropolis Gas Act of 1860, namely, sperm candles of six to the pound, each burning 120 grains per hour. Two of these candles shall be used together. Each testing shall consist of ten observations of the photometer made at intervals of one minute. The average of each set of observations is to be taken as representing the illuminating power for that testing.

The rate of burning of the gas in each burner shall be 5 cubic feet per hour, a rate of consumption which is shown by the long hand of the meter making exactly one revolution per minute for several minutes consecutively.

The candles are to be lighted at least ten minutes before the beginning of each testing, so as to have attained their normal rate of burning. Before and after making each testing the gas examiner shall weigh the candles, and if the rate of consumption per candle shall have not exceeded 126 grains per hour, or fallen short of 114 grains per hour, he shall make, and record in a book to be kept for the purpose, the calculations requisite to neutralize the effects of this difference. If the rate of consumption shall have varied from the prescribed rate beyond the above-named limits, the testing is to be rejected, and a fresh testing made.

Instead of weighing the candles, the gas examiner may observe the time in which 40 grains are burnt. This must not exceed 10·5 or fall short of 9·5 minutes.

The gas examiner shall, at least once a week, compare the meter clock at each testing place.

At the time of each testing the gas examiner shall observe and record the temperature of the gas, as shown by the thermometers attached to the meters, and also the height of the barometer. The volumes of the gas operated upon during the testing may be corrected from these data (the standard of comparison being, for the barometer, 30 inches, and for the thermometer 60 degrees) by means of the tables (see page 312). Suppose, for example, the thermometer stands at 54 degrees, and the barometer at 30·3 inches; multiply the quantity of gas consumed by the corresponding *tabular number*, the product will be the corrected volume of the gas, *i.e.*, the volume the gas would have occupied when measured over water at the standard temperature and pressure. Thus: Volume of gas consumed, 5 cubic feet. Tabular number for thermometer and barometer, 1·025. Then 1·025 × 5 = 5·125, the corrected volume. The gas examiner shall record his observations and calculations for determining illuminating power in the form described on page 314.

The calculations for working out the corrections, &c., for the illuminating power of the gas, proceed in the following manner:—Add the observations together, and divide the sum by 10, to get the average; then, as two candles were used, multiply by two, to get the illuminating power of the gas if tried against one candle. Then, as the standard rate of consumption of the candles, viz., 120 grains, is to the average number of grains consumed by each per hour, so is the above obtained number to the actual illuminating power. Finally make the correction for temperature and pressure by dividing the illuminating power by the tabular number; for example, taking the tabular number as 1·025:—

OBSERVATIONS.

1st minute	7·8
2nd "	7·8
3rd "	8·1
4th "	8·2
5th "	8·3
6th "	8·5
7th "	8·6
8th "	8·4
9th "	8·3
10th "	8·6

10)82·6

Average by 2 candles = 8·26
2

Average by 1 candle = 16·52

Average by 1 candle . . = 16·52
Consumption by 1 candle per hour 123 grains
4956
3304
1652
Standard consumption ·120)203196
Correction for temp. and press. ·1025)16933(16·5 corrected Illum.
1025 Power in candles.
6683
6150
5330

The foregoing calculation can be shortened as follows, which the form prescribed on page 308.

Average by 2 candles . . . 8·26
Consumption by 2 candle in 10 minutes 41 grains
826
3304
2)33866
Tabular number ·1025)16933(16·5 = corrected Illum.
1025 Power in candles.
6683
6150
5330

The Standard Burner.—The burner which has been adopted as the Standard burner for testing common gas was designed by Mr. Sugg, and was called by him "Sugg's London Argand, No. 1."

An illustration is appended (Fig. 71, p. 311), in which A represents a supply-pipe in section, half size; B the gallery; C the cone; D the steatite chamber; E the chimney.

The following are the dimensions of those parts of the burner upon which its action depends :—

	Inch.
Diameter of supply-pipes . . .	0·08
External diameter of annular steatite chamber	0·84
Internal diameter of annular steatite chamber	0·48
Number of holes	24
Diameter of each hole	0·045
Internal diameter of cone—	
At the bottom	1·5
At the top	1·08
Height of upper surface of cone and of steatite chamber above floor of gallery	0·75
Height of glass chimney . . .	6
Internal diameter of chimney . .	1⅞

The Standard burner for testing cannel gas is a steatite batswing burner, consisting of a cylindrical stem, the top of which is divided by a slit of uniform width. The burner is of the following dimensions :—

	Inch.
External diameter of top of stem .	0·31
Internal diameter of stem . . .	0·17
Width of slit	0·02
Depth of slit	0·15

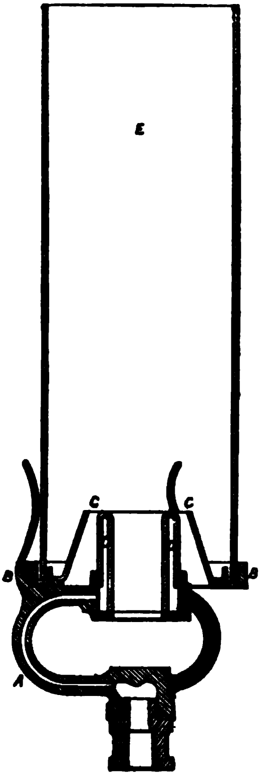

FIG. 71.

On the two following pages will be found a table to facilitate the correction of the volume of gas at different temperatures and under different atmospheric pressures, and at p. 314 is given the form of report to be made by the gas examiner :—

TABLE TO FACILITATE THE CORRECTION OF THE VOLUME OF GAS AT DIFFERENT

BAR.	THER. 40°	42°	44°	46°	48°	50°	52°	54°	56°	58°	60°
28·0	·979	·974	·970	·965	·960	·956	·951	·946	·942	.937	·932
28·	·983	·978	·973	·969	·964	·959	·955	·951	·945	·941	·936
28·	·986	·981	·977	·972	·967	·963	·958	·953	·949	·944	·939
28·3	·990	·985	·980	·976	·971	·966	·961	·957	·952	·947	·942
28·4	·993	·988	·984	·979	·974	·970	·965	·960	·955	·951	·946
28·5	·997	·992	·987	·983	·978	·973	·968	·964	·959	·954	·949
28·6	1·001	·995	·991	·986	·981	·977	·972	·967	·962	·958	·953
28·7	1·004	·999	·994	·990	·985	·980	·975	·970	·966	·961	·956
28·8	1·007	1·003	·998	·993	·988	·984	·979	·974	·969	·964	·959
28·9	1·011	1·006	1·001	·997	·992	·987	·982	·977	·973	·968	·963
29·0	1·014	1·010	1·005	1·000	·995	·990	·986	·981	·976	·971	·966
29·1	1·018	1·013	1·008	1·004	·999	·994	·989	·984	·979	·975	·969
29·2	1·021	1·017	1·012	1·007	1·002	·997	·992	·988	·982	·978	·973
29·3	1·025	1·020	1·015	1·011	1·006	1·001	·996	·991	·986	·981	·976
29·4	1·028	1·024	1·019	1·014	1·009	1·004	·999	·995	·990	·985	·980
29·5	1·032	1·027	1·022	1·018	1·013	1·008	1·003	·998	·993	·988	·983
29·6	1·036	1·031	1·026	1·021	1·016	1·011	1·006	1·001	·996	·992	·986
29·7	1·039	1·034	1·029	1·025	1·019	1·015	1·010	1·005	1·000	·995	·990
29·8	1·043	1·038	1·033	1·028	1·023	1·018	1·013	1·008	1·003	·998	·993
29·9	1·046	1·041	1·036	1·031	1·026	1·022	1·017	1·012	1·007	1·002	·997
30·0	1·050	1·045	1·040	1·035	1·030	1·025	1·020	1·015	1·010	1·005	1·000
30·1	1·053	1·048	1·043	1·038	1·033	1·029	1·024	1·019	1·014	1·009	1·003
30·2	1·057	1·052	1·047	1·042	1·037	1·032	1·027	1·022	1·017	1·012	1·007
30·3	1·060	1·055	1·050	1·045	1·040	1·036	1·030	1·025	1·020	1·015	1·010
30·4	1·064	1·059	1·054	1·049	1·044	1·039	1·034	1·029	1·024	1·019	1·014
30·5	1·067	1·062	1·057	1·052	1·047	1·042	1·037	1·032	1·027	1·022	1·017
30·6	1·071	1·066	1·061	1·056	1·051	1·046	1·041	1·036	1·031	1·026	1·020
30·7	1·074	1·069	1·064	1·059	1·054	1·049	1·044	1·039	1·034	1·029	1·024
30·8	1·078	1·073	1·068	1·063	1·058	1·053	1·048	1·043	1·037	1·032	1·027
30·9	1·081	1·076	1·071	1·066	1·061	1·056	1·051	1·046	1·041	1·036	1·031
31·0	1·085	1·080	1·075	1·070	1·065	1·060	1·055	1·049	1·044	1·039	1·034

⁂ The numbers in the above table have been re-calculated from the formula
the temperature on the Fahrenheit scale, and *a* the tension of aqueous vapour at *t*°.
volume at 60° and 30 inches pressure, V = *v n*.

TEMPERATURES AND UNDER DIFFERENT ATMOSPHERIC PRESSURES.

BAR.	THER. 62°	64°	66°	68°	70°	72°	74°	76°	78°	80°	82°	84°
28·0	·927	·922	·917	·912	·907	·902	·897	·892	·887	·881	·875	·870
28·1	·930	·926	·921	·916	·911	·905	·900	·895	·890	·884	·879	·873
28·2	·934	·929	·924	·919	·914	·909	·904	·898	·893	·887	·882	·876
28·3	·937	·932	·928	·922	·917	·912	·907	·902	·896	·891	·885	·880
28·4	·941	·936	·931	·926	·921	·915	·910	·905	.900	·894	·888	·883
28·5	·944	·939	·934	·929	·924	·919	·914	·908	·903	·897	·892	·886
28·6	·947	·943	·938	·932	·927	·922	·917	·912	·906	·901	·895	·889
28·7	·951	·946	·941	·936	·931	·925	·920	·915	·909	·904	·898	·893
28·8	·954	·949	·944	·939	·934	·929	·924	·918	·913	·907	·901	·896
28·9	·958	·953	·948	·942	·937	·932	·927	·921	·916	·910	·905	·899
29·0	·961	·956	·951	·946	·941	·935	·930	·925	·919	·914	·908	·903
29·1	·964	·959	·954	·949	·944	·939	·933	·928	·923	·917	·911	·906
29·2	·968	·963	·958	·952	·947	·942	·937	·931	·926	·920	·914	·909
29·3	·971	·966	·961	·956	·950	·945	·940	·935	·929	·923	·918	·912
29·4	·975	·969	·964	·959	·954	·949	·943	·938	·932	·927	·921	·915
29·5	·978	·973	·968	·962	·957	·952	·947	·941	·936	·930	·924	·919
29·6	·981	·976	·971	·966	·960	·955	·950	·944	·939	·933	·927	·922
29·7	·985	·980	·974	·969	·964	·959	·953	·948	·942	·937	·931	·925
29·8	·988	·983	·978	·972	·967	·962	·957	·951	·946	·940	·934	928
29·9	·991	·986	·981	·976	·970	·965	·960	·954	·949	·943	·937	·932
30·0	·995	·990	·985	·979	·974	·968	·963	·958	·952	·946	·941	·935
30·1	·998	·993	·988	·983	·977	·972	·966	·961	·955	·950	·944	·938
30·2	1·002	·996	·991	·986	·980	·975	·970	·964	·959	·953	·947	·941
30·3	1·005	1·000	·995	·989	·984	·978	·973	·968	·962	·956	·950	·945
30·4	1·008	1·003	·998	·993	·987	·982	·976	·971	·965	·959	·954	·948
30·5	1·012	1·006	1·001	·996	·990	·985	·980	·974	·969	·963	·957	·951
30·6	1·015	1·010	1·005	·999	·994	·988	·983	·977	·972	·966	·960	·954
30·7	1·018	1·013	1·008	1·003	·997	·992	·986	·981	·975	·969	·963	·957
30·8	1·022	1·017	1·011	1·006	1·000	·995	·990	·984	·978	·972	·967	·961
30·9	1·025	1·020	1·015	1·009	1·004	·998	·993	·987	·982	·976	·970	·964
31·0	1·029	1·023	1·018	1·013	1·007	1·002	·996	·991	·985	·979	·973	·967

$n = \frac{17{\cdot}64\,(h - a)}{460 + t}$, where h is the height of the barometer in inches, t

If v is any volume at t° and h inches pressure, and V the corresponding

STATEMENT OF TESTINGS FOR ILLUMINATING POWER.

Date ____________________ 18

Barometer ________ Thermometer ________ Tabular Number ________

Hour.			
	Consumption of sperm by two candles in ten min.	Consumption of sperm by two candles in ten min.	Consumption of sperm by two candles in ten min.
	Observations.	*Observations.*	*Observations.*
Observations taken at intervals of one minute.	1st min.	1st min.	1st min.
	2nd ,,	2nd ,,	2nd ,,
	3rd ,,	3rd ,,	3rd ,,
	4th ,,	4th ,,	4th ,,
	5th ,,	5th ,,	5th ,,
	6th ,,	6th ,,	6th ,,
	7th ,,	7th ,,	7th ,,
	8th ,,	8th ,,	8th ,,
	9th ,,	9th ,,	9th ,,
	10th ,,	10th ,,	10th ,,
Here multiply the number obtained by the number of grains consumed by the two candles in ten minutes, and divide by 2. Or divide by the number of minutes in which 40 grs. were consumed.	2)	2)	2)

Illum. Power at . . .

,, ,, . . .

,, ,, . . .

Tabular number) (= *Corrected* Average Illum. Power for the day.

Divide the average Illuminating Power by the tabular number corresponding to Mean Temperature and Pressure: the quotient will be the corrected Illuminating Power for the day.

Average Illum. Power uncorrected . . . } = ____________ Candles.

____

Gas Examiner.

Instructions of the London Gas Referees as to the Times and Mode of Testing for Purity.—The testings for purity shall extend over twenty hours of each day, and shall be made upon ten cubic feet of gas, which shall be tested successively for each of the following impurities:—

I. *Sulphuretted Hydrogen.*—The gas shall be passed as it leaves the service-pipe through an apparatus (Fig. 72) in which are suspended slips of bibulous paper, impregnated with basic acetate of lead.

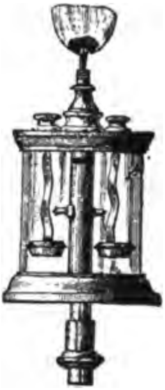

FIG. 72.

The test-paper from which these slips are cut is to be prepared from time to time by moistening sheets of bibulous paper with a solution of one part of sugar of lead in eight or nine parts of water, and holding each sheet while still damp over the surface of a strong solution of ammonia for a few moments. As the paper dries all free ammonia escapes.

If any discolouration of the slip of test-paper is found to have taken place, this is to be held conclusive as to the presence of sulphuretted hydrogen in the gas. Fresh test-slips are to be placed in the apparatus every day.

In the event of any impurity being discovered, one of the test-slips shall be placed in a stoppered bottle and kept in the dark at the testing-place; the remaining slips shall be forwarded with the daily report.

II. *Ammonia.*—The gas which has been tested for the sulphuretted hydrogen shall pass next through an apparatus consisting of a glass cylinder filled with glass beads, which have been moistened with a measured quantity of standard sulphuric acid. A set of burettes, properly graduated, is provided.

The maximum amount of ammonia allowed is 4 grains per 100 cubic feet of gas, and the testings shall be made so as to show the exact amount of ammonia in the gas.

Two test-solutions are to be used—one consisting of dilute sulphuric acid of such strength that 25 measures (septems) will neutralize 1 grain of ammonia; the other a weak solution of ammonia, 100 measures of which contain 1 grain of ammonia.

The correctness of the result to be obtained depends upon the fulfilment of two conditions:—

1. The preparation of test-solutions having the proper strength.

2. The accurate performance of the operation of testing.

To prepare the test-solutions the following processes may be used by the gas examiner:—

Measure a gallon of distilled water into a clean earthenware jar, or other suitable vessel. Add to this 94 septems of pure concentrated sulphuric acid, and mix thoroughly. Take exactly 50 septems of the liquid and precipitate it with barium chloride in the manner prescribed for the sulphur test. The weight of barium sulphate which 50 septems of the test-acid should yield is 13·8 grains. The weight obtained with the dilute acid prepared as above will be somewhat greater, unless the sulphuric acid used had a specific gravity below 1·84.

Add now to the dilute acid a measured quantity of water, which is to be found by subtracting 13·8 from the weight of barium sulphate obtained in the experiment, and multiplying the difference by 726. The resulting number is the number of septems of water to be added.

If these operations have been accurately performed, a second precipitation and weighing of the barium sulphate obtainable from 50 septems of the test-acid will give nearly the correct number of 13·8 grains. If the weight exceeds 13·9 grains, or falls below 13·7 grains, more water or sulphuric acid must be added, and fresh trials made, until the weight falls within these limits. The test-acid thus prepared should be transferred at once to stoppered bottles, which have been well drained and are duly labelled.

To prepare the standard solution of ammonia, measure out as before a gallon of distilled water, and mix with it 50 septems of strong solution of ammonia (sp. gr. 0·88). Try whether 100 septems of the test-alkali thus prepared will neutralize 25 of the test-acid, proceeding according to the directions given subsequently as to the mode of testing. If the acid is just neutralized by the last few drops, the test-alkali is of the required strength. But if not, small additional quantities of water, or of strong ammonia solution, must be added, and fresh trials made, until the proper strength has been attained. The bottles in which the solution is stored should be filled nearly full and well stoppered.

The mode of testing is as follows:—Take 50 septems of the test-acid (which is greatly in excess of any quantity of ammonia likely to be found in the gas) and pour it into the glass cylinder, so as to well wet the whole interior surface, and also the glass beads. Connect one terminal tube of the cylinder with the gas supply and the other with the meter, and make the gas pass at the rate of about half a cubic foot per hour. Any ammonia that is in the gas will be arrested by the sulphuric acid, and a portion of the acid (varying with the quantity of ammonia in the gas) will be neutralized thereby. At the end of each period of testing, wash out the glass cylinder and its contents with distilled water, and collect the washings in a glass vessel. Transfer one-half of this liquid to a separate glass vessel, and add a quantity of a neutral solution of hæmatoxylin or litmus just sufficient to colour the liquid. Then pour into the burette 100 septems of the test-alkali, and gradually drop this solution into the measured quantity of the washings collected, stirring constantly. As soon as the colour changes (indicating that the whole of the sulphuric acid has been neutralized), read off the quantity of liquid remaining in the burette. To find the number of grains of ammonia in 100 cubic feet of the gas, multiply by 2 the number of septems of test-alkali remaining in the burette, and move the decimal point one place to the left.

The remaining half of the liquid is to be preserved for a week in a bottle duly labelled.

III.—*Sulphur Compounds other than Sulphuretted Hydrogen.* —The gas which has been tested for sulphuretted hydrogen and ammonia shall pass next through a meter, by means of which the rate of flow can be adjusted to half a cubic foot per hour, and which is provided with a self-acting movement for shutting off the gas when ten cubic feet have passed.

The testing shall be made in a room where no gas is burnt other than that which is being tested for sulphur and ammonia.

The apparatus to be employed is represented by the diagram (Fig. 58), and is of the following description:—The gas is burnt in a small Bunsen burner with steatite top, which is mounted on a short cylindrical stand, perforated with holes for the admission of air, and having on its upper surface a deep circular channel to receive the wide end of a glass trumpet-tube. On the top of the stand, between the narrow stem of the burner and the surrounding glass trumpet-tube, are to be placed pieces of commercial sesqui-carbonate of ammonia weighing in all about 2 ounces.

The products both of the combustion of the gas and of the gradual volatilization of the ammonia salt go upwards through the trumpet-tube into a vertical glass cylinder, packed with balls of glass, to break up the current and promote condensation. From the top of the cylinder there proceeds a long glass pipe or chimney, serving to effect some further condensation, as well as to regulate the draught and afford an exit for the uncondensable gases. In the bottom of the cylinder is fixed a small glass tube, through which the liquid (formed during the testing) drops into a beaker beneath.

The following cautions are to be observed in selecting and setting up the apparatus:—

> See that the inlet-pipe fits gas-tight into the burner, and that the holes in the circular stand are clear. If the burner gives a luminous flame remove the top piece, and, having hammered down gently the nozzle of soft metal, perforate it afresh, making as small a hole as will give passage to half a cubic foot per hour at a convenient pressure.

> See that the tubulure of the condenser has an internal diameter of not less than ¾ inch, and that its outside is smooth and of the same size as the small end of the trumpet-tube.
>
> See that the short piece of india-rubber pipe fits tightly both to the trumpet-tube and to the tubulure of the condenser.
>
> The small tube at the bottom of the condenser should have its lower end contracted, so that when in use it may be closed by a drop of water.
>
> The india-rubber pipe at the lower end of the chimney-tube should fit into, and not simply rest upon, the mouth of the condenser, and the upper extremity of this tube may with advantage be given a downward curvature.

At the end of each period of testing, the cylinder and trumpet-tube are to be well washed out with distilled water. Fresh pieces of sesqui-carbonate of ammonia are to be used each day.

The gas examiner shall then proceed as follows:—

The liquid in the beaker and the water used in washing out the apparatus shall be put into the same vessel, well mixed, and measured. One-half of the liquid so obtained is to be set aside, and preserved for a week, properly labelled, in case it should be desirable to verify the correctness of the testing.

The remaining half of the liquid is to be put into a flask, or beaker covered with a large watch-glass—treated with hydrochloric acid in sufficient quantity to leave an excess of acid in the solution—and then raised to the boiling point. An excess of a solution of barium chloride is now to be added, and the boiling continued for five minutes. The vessel and its contents are to be allowed to stand till the barium sulphate settles at the bottom of the vessel, after which the clear liquid is to be as far as possible poured off through a paper filter. The remaining liquid and barium sulphate are then to be poured on to the filter, and the latter well washed with hot distilled water. (In order to ascertain whether every trace of barium chloride and ammonium chloride has been removed, a small quantity of the washings from the filter should be placed in a test-tube, and a drop of a solution of silver nitrate added; should the liquid, instead of remaining perfectly clear, become cloudy, the washing must be continued until, on repeating the test, no cloudiness is produced.) Dry the filter with its contents, and transfer it into a weighed platinum crucible. Heat the crucible over a lamp, increasing the temperature gradually, from the point at which the paper begins to char, up to bright redness. When no black particles remain, allow the crucible to cool; place it when nearly cold in a desiccator over strong sulphuric acid, and again weigh it. The difference between the first and second weighings of the crucible will give the number of grains of barium sulphate. Multiply this number by 11 and divide by 4; the result is the number of grains of sulphur in 100 cubic feet of the gas.

This number is to be corrected for the variations of temperature and atmospheric pressure in the manner indicated under the head of Illuminating Power (see *post*), with this difference, that the readings of the barometer and thermometer are to be taken for the day on which the testing commenced and also the day on which it closed; and the mean of the two is to be used.

This correction may be made most simply and with sufficient accuracy in the following manner:—

When the tabular number is between 955-965, 966-975, 976-985, 986-995, increase the number of grains of sulphur by $\frac{4}{100}$ths, $\frac{3}{100}$ths, $\frac{2}{100}$ths, $\frac{1}{100}$th.

When the tabular number is between 996-1005, no correction need be made.

When the tabular number is between 1006-1015, 1016-1025, 1026-1035, diminish the number of grains of sulphur by $\frac{1}{100}$th, $\frac{2}{100}$ths, $\frac{3}{100}$ths.

EXAMPLE:	Barometer (mean) . .	29·4
	Thermometer (mean) . . .	58
	Tabular number	985

Grains of barium sulphate from 5 cubic feet of gas	4·3
Multiply by 11, and divide by 4	11
	4)47·3
Grains of sulphur in 100 cubic feet of gas (uncorrected) . .	11·82
Add $11·8 \times \frac{2}{100} =$	·24
Grains of sulphur in 100 cubic feet of gas (corrected) . . .	12·06

[Result: 12·1 grains.]

As to the Maximum Amounts of Impurity in each Form with which the Gas shall be allowed to be Charged.

Sulphuretted Hydrogen.—By the Acts of Parliament all gas must be wholly free from this impurity.

Ammonia.—The maximum amount of this impurity shall be 4 grains per 100 cubic feet of gas.

Sulphur Compounds other than Sulphuretted Hydrogen.—The maximum amount of sulphur with which gas shall be allowed to be charged shall be 22 grains of sulphur in every 100 cubic feet of gas.

Method of Valuing Coal for Gas Making.—An elementary analysis of a coal is not by any means a good criterion as to the amount and quality of gas that can be produced from it. In large gas-works there is generally a miniature gas-works which is used to value various coals for their gas, tar, and ammoniacal liquor. Newbigging uses an apparatus capable of distilling or carbonizing $\frac{1}{1000}$ of a ton, = 2·24 lbs., at a time. Fig. 73 is a sketch of such an apparatus.

It consists of a cast-iron D-shaped retort, A, of ¼-inch metal, 5 inches wide, 4½ inches high inside, 2 feet 3 inches long outside; an ascension-pipe, B, of 2-inch wrought iron, connected with a 1½-inch wrought iron tube, C, to the condenser, D, consisting of twelve vertical 1½-inch wrought tubes 3 feet 6 inches long; these are attached to the waster, E, 1 foot long, 6 inches wide, and 6 inches deep, and connected to the purifier, E, which is 1 foot 2 inches square, 12 inches deep, and contains two trays for lime; the gas-holder, G, has a capacity of 12 cubic feet, and has a graduated scale attached so that the volume of gas produced can be at once observed.

On starting a test of a sample of coal it is of course very essential that an average sample is taken, and small pieces, about the size of a walnut, used. Three quantities of 2·24 lbs. each will be found sufficient to give a reliable opinion as to the value of the coal. The retort must be first got up to a bright red heat before the charge is introduced, and must be maintained at this temperature during the whole of the distillation.

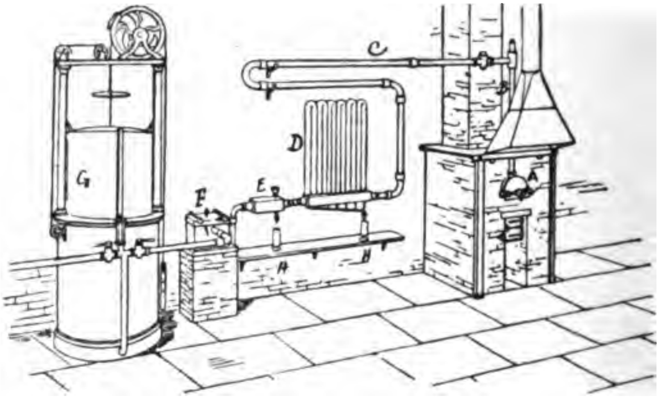

FIG. 73.

The time required to carbonize each charge of 2·24 lbs. will be from 40 to 60 minutes. The tar and ammoniacal liquor produced are drained out of the condensers and washers into the graduated test-glasses, H H, and measured; after the volume of gas has been read off its illuminating power may be deduced by the method described on page 307. The coke is drawn out of the retorts into a bucket provided with a lid and surrounded with cold water, and when cool its weight is ascertained and calculated per ton of coal.

The following are results obtained for various kinds of coal·—

Name of coal.	Cubic feet of gas per ton.	Illuminating power —candles.	Coke per ton of coal in lbs.	Ash in coal per cent.
Ebbw Vale (Graig Fawr).	9,800	15·50	1,558	2·75
Sunybank Cannel	12,000	36·50	1,086	15·5
Boghead „	14,550	30·6	1,015	7·5
Newcastle „	11,560	28·0	1,350	9·1
Adwalton „	10,850	20·2	1,300	5·8
Wigan „	11,200	22·5	1,420	5·5
Llantwit (bituminous)	9,708	18·30	1,345	5·0
Aberbeeg „	8,886	17·10	1,590	4·5
Abercarn „	12,200	16·00	1,410	6·0
Radstock „	10,250	18·25	1,310	5·85
Caleford „	9,500	16·15	1,355	7·76
Hartley „	10,500	12·50	1,435	3·50
New Pelton „	10,480	12·85	1,558	2·75
Hindley Field „	10,750	15·50	1,320	5·25
Lydney „	9,500	15·55	1,350	6·25
Thorncliffe „	10,500	16·50	1,435	3·65
Gosforth „	10,025	13·45	1,450	5·50

Ammoniacal Liquor.

The ammoniacal liquor of the gas-works, which derives its ammonia from the nitrogen present in the coal from which it is distilled, is a complicated solution containing a number of ammoniacal compounds, of which the following is a list:—

(*a*) VOLATILE AT ORDINARY TEMPERATURES.

Free ammonia.
Ammonium carbonates (mono, sesqui, and bi).
„ sulphide.
„ bisulphide.
„ acetate.
„ cyanide.

(*b*) FIXED AT ORDINARY TEMPERATURES.

Ammonium sulphate.
„ sulphite.
„ hyposulphite.
„ thiocarbonate.
„ chloride.
„ sulphocyanate.
„ ferrocyanide.

together with smaller portions of tarry matters, such as phenol, benzol, &c., pyridine, salts, &c.

Gas liquor is principally used for the production of sulphate of ammonia, much used as a manure, and its value for this purpose depends upon the proportion of total ammonia (free and combined) that it contains. Of the total ammonia from the liquor produced from English coals 15 to 20 per cent. is in the "fixed" state.

The following interesting table by Gerlach shows the amount of total ammonia contained in the ammoniacal liquors from various German and English coals:—

Coal Distilled.	grms. NH$_3$ per 100 cc.	degs. Baumé.	Gas Works.
German Coals.			
Zwickau coal and Bohemian lignite	0·908	1·66	Leipzig
Zwickau and Burgk coal	0·944	1·5	Dresden
Burgk coal	1·862	2·8	Freiberg
Ruhr coal	1·812	2·1	Bonn
,,	1·292	1·75	Eupen
,,	1·826	2·25	Cologne
,,	1·278	1·5	Dortmund
Saar coal	1·574	—	Augsberg
,,	1·523	2·5	Treves
,,	1·870	3·2	Zurich
Silesian coal	2·339	3·0	Berlin
,, ,,	1·290	—	Pest
,, ,, and Bohemian lignite	1·030	2·0	Prague
English Coals.			
Cannel coal: boghead	2·881	3·75	Hamburg
English coal	3·514	4·0	Stettin
,, ,,	2·659	3·0	St. Petersburg
,, ,,	2·244	3·3	Trieste
,, ,,	2·142	3·5	Stralsund
Leverson Wallsend	2·366	4·0	Altona
English coal	2·407	4·0	Copenhagen
New Pelton: Ravensworth	1·785	2·75	Riga
Old Pelton Main	1·717	3·0	Königsberg
English coal	2·966	3·0	Stettin
Old Pelton Main: Leverson Wallsend	1·345	3·0	Dantzic

The Valuation of Ammoniacal Liquor.—We shall here only make a determination of the free and combined ammonia with the amount of lime required and its cost for expelling the combined ammonia. A method much used for valuing liquors is to obtain the specific gravity of them, and by referring to a table the percentage of ammonia is obtained; but owing to the difference in density of the various ammonium salts present, this method might be considered practically worthless. The following table by Siedel shows the differences in the percentage of ammonia in different liquors of the same specific gravity at 15° C.:—

Degrees, Baumé .	2	2·5	3	3·5	4	4·5	5	6
Specific gravity . .	1·0138	1·0163	1·0208	1·0249	1·0280	1·0316	1·0352	1·0426
Per cent. NH_3	1·16	1·30						
	1·42	1·43						
	1·50	1·63	1·63					
	1·77	1·77	1·76					
		1·98	1·90	1·87				
		2·18	2·10	2·00				
		2·65	2·38	2·24				
			2·45	2·40	2·55			
				2·72	2·72	2·79		
					2·90	2·85		
					3·40	3·06		
						3·40		
						3·53	3·67	3·74

The following is a good method for the determination of the amount of the free and combined ammonia present in gas liquors. The specific gravity is first taken at 15° by hydrometer.

Determination of "Free" Ammonia.—Accurately measure out 20 cc. of the sample, pour into the flask A, Fig. 74, holding about 300 cc., dilute with about 100 cc. of distilled water, fix to condensing arrangement as shown in the sketch, and keep the tap B closed. E is a tube filled with glass wool, and serves to trap any traces of ammonia that might otherwise escape. When all is ready fixed for distillation, 30 cc. of *standard* E H_2SO_4 are run into D through the tube E, followed by a few drops of litmus solution. The glass wool will thus be coloured red. If any of the ammonia escapes (which rarely happens) during the distillation, the stratum of glass wool would be completely coloured blue. Apply a gentle heat to A until it gently boils, and keep at this temperature until about 60 cc. have collected in D. The tap B is now opened and all acid washed out of E into D. It now becomes necessary to ascertain how much of the acid has been neutralized by the "free" ammonia distilled off; for this purpose detach D, gently boil to eliminate any CO_2, fill a burette with *standard* E sodic hydrate, and run this into it gradually until the last drop added turns the solution from red to blue. For every cc. of E H_2SO_4 neutralized by the sample there is ·017 grm. of NH_3 in solution.

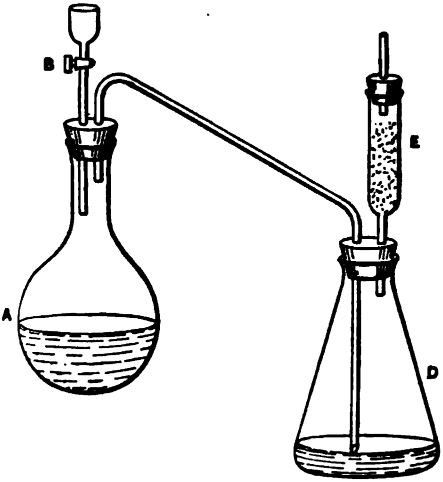

FIG. 74.

Combined Ammonia.—The flask D is now swilled out and again attached to E and A, and 20 cc. of standard E H_2SO_4 coloured with litmus are poured through E into D; 40 cc. of E NaHO (recently boiled to eliminate any NH_3 present) are now run into the residual solution in A through B, and the distillation proceeded with as before, the solution being reduced nearly to dryness. The free acid in D is titrated with standard E NaHO as before. The number of cc.'s of acid neutralized × ·017 = grms. of *combined* ammonia in 20 cc. of sample.

Example.—A sample of ammoniacal liquor had a specific gravity of 1·026 at 60° F.

Free Ammonia.—20 cc. of sample were taken, diluted, and distilled into 30 cc. of standard E H_2SO_4. On titration with E NaHO 11·6 cc. were required to produce blue tint.

30 — 11·6 = 18·4 cc. E H_2SO_4 neutralized.
1 cc. of E H_2SO_4 = ·017 grm. NH_3.
18·4 × ·017 = ·3128 grm. NH_3 in 20 cc.
20 cc. of liquor = 20 × 1·026 (sp. gr.) = 20·52 grms.
20·52 : 100 : : ·3128 : *x* — = 1·5244 per cent. "free" ammonia.
1 gallon of liquor weighs 10 × 1·026 = 10·26 lbs.
103 : 10·26 : : 1·5244 : *x* — = 0·1564 lbs. free NH_3 per gallon.

Combined Ammonia.—The residual solution from the estimation of the "free" ammonia was distilled with sodic hydrate into 20 cc. of standard E H_2SO_4, and on titrating the excess of acid back with E NaHO, 11·7 cc. were required to produce blue tint.

20 — 11·7 = 8·3 cc. E H_2SO_4 used.
8·3 × ·017 = ·1411 grm. combined NH_3 in 20 cc. (= 20·52 grms.) of liquor.
20·52 : 100 : : ·1411 : *x* — = 0·6876 per cent. combined ammonia.

TOTAL AMMONIA.

1·5244 + ·6876 = 2·212 per cent. total ammonia.
100 : 10·26 : : 2·212 : *x* = ·22695 lbs. of total NH_3 in a gallon of liquor.

Now every 34 parts of ammonia (NH_3) form 132 parts of ammonic sulphate $(NH_4)_2 SO_4$ then

$$34 : ·22695 : : 132 : x = 0·8811 \text{ lb. of } Am_2SO_4$$

could be produced from one gallon of liquor; and $\frac{2240}{·8811} = 2542$

gallons of liquor required, to produce one ton of pure sulphate of ammonia.

Quantity and price of lime required to expel the combined ammonia from 1,000 gallons of liquor. Say 90 per cent. lime is 22s. per ton.

Now every 34 parts of combined ammonia require 56 parts of pure lime (CaO) for its expulsion.

1,000 gallons of the liquor weigh 10,260 lbs., and the number of lbs. of combined ammonia in 1000 gallons is—

$$100 : 10{,}260 :: \cdot 6876 : x - = 70\cdot 55 \text{ lbs.}$$

$$\because \text{ lbs. of pure lime required } - = \frac{70\cdot 55 \times 56}{34} = 116\cdot 2 \text{ lbs.}$$

$$,, \quad 90\,\% \quad ,, \quad ,, \quad = \frac{100 \times 116\cdot 2}{90} = 129 \text{ lbs.} = 1\text{s. } 4\text{d.}$$

per 1,000 gallons.

Assay of Sulphate of Ammonia (Am_2SO_4).

The value of commercial sulphate of ammonia in this country, is deduced from the percentage of ammonia present, and in Germany from the percentage of nitrogen. The pure salt contains 21·21 per cent. of nitrogen, equivalent to 25·75 per cent. of ammonia (NH_3). The specification for sulphate in this country enacts that it should contain 23 per cent. of combined ammonia; but the *commercial* article often contains between 24 and 25 per cent. The analysis of sulphate of ammonia should include the determination of water, combined ammonia, free sulphuric acid, and any sulphocyanates.

Estimation of Water.—10 grms. of the sample are weighed into a tared beaker and dried in the water oven until no further loss occurs; the loss is taken as water.

Estimation of Free Sulphuric Acid.—Weigh out 5 grms. of the sample into a 150 cc. beaker, dissolve in 100 cc. of distilled water, boil for a few minutes to expel any CO_2, cool, add a few drops of litmus solution, and run in standard E sodic hydrate from a burette until the last drop added turns the solution from red to blue. Note volume required. Each cc. of E $NaHO$ = ·049 grm. of free sulphuric acid present.

Estimation of Combined Ammonia.—The method adopted for the estimation of the combined ammonia in gas liquor is suitable for its determination in sulphates. 10 grms. of the averaged sample are weighed into a beaker, dissolved in water, diluted to 250 cc. at 15° C., and thoroughly mixed. 50 cc. of this solution (= 2 grms. sample) are now poured into the flask A, Fig. 47, and 35 cc. of *standard* E H_2SO coloured with litmus, added through E into the flask D, and attached ready for distillation. 5 grms. of sodic hydrate dissolved in 50 cc. of water and boiled to expel any NH_3 present, are now poured into A through the tap B, which is afterwards closed, and the distillation proceeded with, which should take about two hours, the solution being prevented from getting too low in A by frequent additions of water through B. After it is assured that all the ammonia has been expelled, the tube E is washed into D, and the excess of acid therein titrated with standard E $NaHO$; each cc. of acid neutralized = ·017 grm. of ammonia.

Estimation of Sulphocyanates.—Owing to defective manufacture, small quantities of sulphocyanic acid often find its way into the finished product, which is supposed to have a very detrimental influence on plant life, and thus materially diminishes its value as a manure. A rough method much used for testing for sulphocyanates, is to place a small heap of 5 grms. of the sample on a sheet of white paper, and drop on it a solution of ferric chloride until the whole becomes saturated with it, when, if any sulphocyanates be present, a more or less red coloration will be produced, owing to the formation of ferric sulphocyanate. The following reaction takes place:—

$$\underbrace{6\,NH_4CNS}_{\text{Ammonic sulphocyanate.}} + Fe_2Cl_6 = \underbrace{Fe_2\,(CNS)_6}_{\text{Ferric sulphocyanate.}} + 6\,NH_4Cl.$$

If a similar heap of pure sulphate of ammonia, free from sulphocyanate, be placed by the side of the sample, and saturated with ferric chloride solution, a solution of potassic sulphocyanide of known strength can be gradually run on to it, and stirred with a glass rod until it is seen that the tints are near alike; a rough idea is thus formed of the amount present.

The proximate estimation of sulphocyanate in commercial sulphate of ammonia is attended with difficulties, owing to the small quantity usually present, and to the influence of other complicated substances on its estimation by the usual processes. With care, the following colorimetric process based on the above reaction, will give fair results :—

Weigh out 4 grms. of the sample, dissolve in a little water, dilute to exactly 14 cc., filter off 7 cc. (= 2 grms. sample), into a carbon tube (Fig. 9), add 0·5 cc. of 5 E H_2SO_4, and 1 cc. of 5 E Fe_2Cl_6, and thoroughly mix. It now becomes necessary to obtain a standard ferric sulphocyanate solution. Owing to the peculiar tint of a solution of the commercial sulphate, neither an aqueous nor a pure ammonic sulphate solution of the same strength will answer for comparison; the writer finds the best method is to make a *standard* of some of the sample, in which the sulphocyanates have been previously eliminated by evaporating to complete dryness with sulphuric acid. Weigh out 4 grms. of the sample into a small porcelain dish, mix with 1 cc. of 5 E H_2SO_4, and dry in a water oven for a couple of hours. By this means the sulphocyanates will be practically all gone. Dissolve in water, dilute to 10 cc., filter off 5 cc. into a carbon tube, add 1 cc. of 5 E Fe_2Cl_6, and dilute to within 2 cc. of the volume occupied by the sample; now run in 0·2 cc. at a time, from a 2 cc. pipette, graduated in ·05 cc.'s, a solution of $\frac{E}{50}$ potassic sulphocyanate, until the tints are nearly alike, finally adjusting the volumes with water.

$$1 \text{ cc. of } \frac{E}{50} \text{ KCNS} = \cdot 00116 \text{ grm. CNS.}$$

The following is an analysis, by the above processes, of a bad sample of sulphate :—

Water	3·50	per cent.
Combined ammonia	23·45	"
Free sulphuric acid	trace	
Sulphocyanate (CNS) . . .	·09	"
Corresponding to AmCNS . . .	·12	"

Spent Oxide.

The important part played by hydrated peroxide of iron in the absorption of sulphuretted hydrogen and other detrimental constituents of crude coal gas, renders the residual spent oxide a valuable product. With sulphuretted hydrogen the following reaction occurs:—

$$Fe_2O_3 .\ x\ OH_2 + 3\ SH_2 = \underbrace{Fe_2S_3 .\ x\ OH_2}_{\text{Sesquisulphide of Iron.}} + 3\ OH_2.$$

On exposing the sesquisulphide of iron thus produced to the action of the air, peroxide of iron is again produced, while free sulphur is formed thus:—

$$2\ Fe_2S_3 .\ x\ OH_2 + 3\ O_2 = 2\ Fe_2O_3\ x\ OH_2 + 3\ S_2,$$

and the mixture would be capable of absorbing a further quantity of SH_2; the absorption and exposure could be repeated several times, until the oxide contains 55 per cent. of sulphur.

When the spent oxide has completed its duty, it is left exposed to the atmosphere, with frequent stirring up, until completely revivified; it is then assayed for sulphur and sold, the price varying according to the amount of sulphur present.

Estimation of Sulphur.—The recognised method for the determination of sulphur in spent oxide is very simple. All that has to be done is to dissolve the sulphur out of a known weight of the sample by means of pure redistilled disulphide of carbon, which is afterwards distilled in a weighed flask, and the residual sulphur weighed. The following is the manner in which the sulphur is determined: 5 grms. of the averaged sample are weighed into the tube A (Fig. 75), into which a plug of glass wool is fitted in the constriction. It is fitted into the weighed flask B contained in a sand bath, and connected to the bottle G, containing pure disulphide of carbon, by the tube T, and to the condenser D, by means of the tube M. When all is fixed, CS_2 is made to percolate through the sample by blowing into G, through Q. The sulphur is thus dissolved out into B; when B is nearly full the Bunsen, F, is lighted, and the disulphide distilled off through D into the flask E, which is afterwards poured back into G, and the percolation continued several times, until it is assured that the whole of the sulphur is dissolved out. The CS_2 having been finally distilled, the residual sulphur is dried in the water oven and weighed; its weight × 20 = percentage of sulphur.

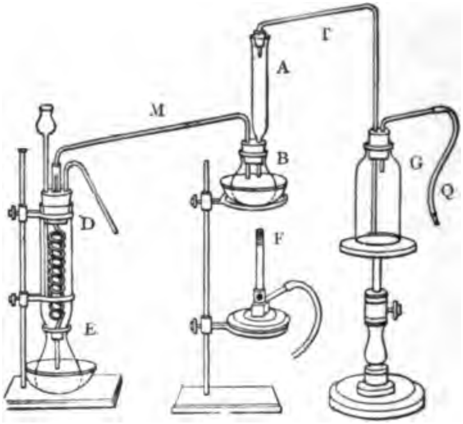

FIG. 75.

Synopsis of the Distillation of Coal Tar.—The following tabular statement shows the manner in which the various products from coal tar are obtained :—

Dehydration, *a.* by standing . . . . .
b. during the heating up . . **Ammoniacal Liquor**
Distillation.
I. *Fraction up to* 170° C. { Ammoniacal liquor
First runnings

rectified yield :—
(1) Product up to 110° C., chemically washed
Distilled by steam, yields, *a* . . . .
b. Weaker benzol goes to 1, 2 . . **90 per cent. Benzol**
(2) Product up to 140° treated like (1), yields *a*
b. . . . . **50 per cent. Benzol**
c. Intermediate fraction is redistilled
d. . . . .
(3) Product up to 170° treated like (1) and (2) **Solvent Naphtha**
yields *a* . . . .
b. . . . . **Burning Naphtha**
c. Residue, goes to II.

II. *Fraction from* 170° to 230°. Middle oil—
washed with caustic soda yields :—
(1) Oil, distilled in the light oil still, yields—
a. Distillate up to 170° goes to (1), (3)
b. „ „ 230°, yields . . . **Napthalene**
c. Residue goes to III.
(2) Alkaline liquor decomposed by CO_2 yields :—
a. Aqueous solution of sodium carbonate, causticised by lime and used over again
b. Crude carbolic acid is purified and yields, *α* . . . . **Carbolic Acid**
β. Waste oils, go back to II.

III. *Fraction from* 230° to 270° = *Heavy oil* (collected till solid matters begin to crystallize).
Can be treated for naphthalene, usually
only employed as . . . . **Creosote Oil**
or else separated into *a* . . .
b . . . **Lubricating Oil**

IV. *Fraction, anthracene oil*
Filtered or cold-pressed yields :—
(1) Oils, are redistilled and yield
a. Solid distillate, treated along with IV. (2)
b. Liquid distillate, goes to III. *b*, or is redistilled.
c. Residue (pitch, coke, &c.)

(2) Residue is hot pressed and yields
  a. Oils, treated like IV. (1)
  b. Crude anthracene, washed with naphtha, &c., yields, *a* . . . . . . **Anthracene**
    β. Solution is distilled, and yields,
      aa. Naphtha used over again for washing
      bb. Phenanthrene, &c., is burnt to . **Lampblack**

V. Pitch. Employed for patent fuel or varnishes, &c. . . . . **Pitch**
  or else distilled, yielding:—
(1) Crude anthracene, treated like IV. (2)
(2) Lubricating oil, goes to III. *a*, resp. III. *b*
(3) Residue . . . . **Coke**

The following table shows the yields, in percentage, of the various products from different tars (Calvert).

	Light oil.	Neutral heavy oil.	Phenol.	Paraffin.	Naphthalene.	Pitch.
Boghead . .	12	30	3	41	—	14
Cannel coal .	9	40	14	—	15	22
Newcastle coal	2	12	5	—	58	23
Staffordshire coal	5	35	9	—	22	29

CREOSOTE.

Creosote as used for the "Pickling" of Timber.

The following is an extract from an interesting report which was made by Dr. Tidy to the Directors of the Gaslight and Coke Company, in August, 1883,* upon this subject:—

"The advantages to be derived from the creosoting process are of a threefold nature:

"1st. *A physical action.*—A very great increased solidity is effected by choking up the pores, thus agglutinating the whole mass of the wood into a more or less solid block. Apart from its rendering the wood more solid, this physical action is important in preventing the subsequent absorption of moisture.

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* *Vide* Lunge's "Coal Tar and Ammonia."

"2nd. *A physiological action.*—The smell of creosote imparted to the wood prevents germinal life, well known to be destructive to timber, from being developed within it. Seeing that the preservation of timber has been effected by such materials as chloride of zinc, sulphate of copper, &c., with greater or less success, and that the action of these bodies must be mainly—although I admit not entirely—dependent on their toxic properties, this physiological action is one of importance. It must be remembered, however, that creosote has the advantage of a well-marked smell, which odour most of the lower animals dislike. In this respect it is superior to the other bodies I have named.

"Further, it is worth pointing out that all the constituents of the coal tar, and not the tar acids only, have a more or less well-marked tarry odour.

"3rd. *A chemical action.*—Respecting the chemical action I would draw attention to the fact that tar acids are not only antiseptic, but that they possess the power of coagulating albumen. It is to this latter action that I shall have to refer later on in this report, as playing an important part, in my opinion, in the preservation of timber.

"Now the following questions arise :—(1st.) Upon what constituents of the creosote does its value specially depend, and what are the relative values of its different constituents? (2nd.) If there be constituents in the creosote, which of themselves possess no special value, do they in any respect lessen the activity of the valuable constituents?

"The importance of considering the precise value of the several constituents of creosote arises as follows :—

"Speaking generally, creosote may be divided into two classes, London and Country creosotes. By London creosote we mean the creosote derived from the tars of London gasworks, the east coast generally, and from the gasworks of towns such as Southampton, Brighton, &c., where the coal employed is Newcastle coal. So far as I am able to learn, the larger proportion of the creosote produced in England is of this character. The two creosotes being very different in their composition, it becomes important to consider them separately.

"The *London* creosote has a somewhat high specific gravity, and contains a comparatively large percentage of naphthalene, and a small percentage (*i.e.*, less than 10 per cent.) of tar acids. Further, it contains a considerable quantity of the heavier portions of the oil, that is, of those portions not volatile at a temperature below 316° C.

"The *country* creosote, on the other hand, has a less specific gravity, and is considerably more fluid than London creosote. It contains considerably less naphthalene than the London creosote, a larger total percentage of tar acids, and a smaller percentage of the heavier portions of the oil present.

"The real question I have had in view in this inquiry being Country creosotes *versus* London creosotes, it became necessary to inquire into the relative values of the heavier portions of the oil, of the naphthalene, and of the tar acids in creosoting.

"The tar acids, in the first instance, effect the coagulation of the albumen of the wood sap. This coagulated albumen mixes with the naphthalene of the creosote, which, so soon as the temperature becomes sufficiently reduced, is redeposited, and forms, along with the heavier portions of the oil, a solid magma within the pores and fibres of the wood. That this formation of solid magma actually occurs, I have convinced myself by numerous microscopic examinations of creosoted timbers.

"The success of the process, therefore, being presumably assisted by the coagulation of the albumen, the question arises, *What quantity of tar acids is necessary to effect this object?*

"There is little doubt in my mind that 2 or 3 per cent. would amply suffice to effect this coagulation of the sap albumen.

"We are now led to consider if any value and, if any, what value is to be ascribed to the tar acids beyond that needed to effect the coagulation of the albumen.

"I am far from prepared to say they are otherwise entirely valueless. Still it is a remarkable fact, which I have over and over again verified, that in the timbers that have been creosoted for a considerable time (say a year), very small quantities indeed (if any) of the free tar acids are to be found.

"I have upon this point instituted a series of examinations of sleepers, obtained from independent sources, and of ages varying from one to twenty years; and it is a fact worth noting that, within a very short time after a sleeper has been in use, the tar acids appear to be entirely dissipated.

"Seeing however that the life of a sleeper is by no means so limited, the facts I have mentioned suffice to show that the action of the tar acids *per se* cannot have any great or permanently preservative influence in creosoting.

"I admit it was natural to suppose that bodies commonly regarded as powerfully antiseptic should have been the active agents in the process. Further, I must admit that it was with such view I commenced this inquiry. My recent investigations, however, have clearly shown that the value of the tar acids in the creosoting process has been greatly over-estimated.

"I am convinced that, so long as the quantity of carbolic acid present in the creosote is sufficient to coagulate the albumen of the wood sap, it is also sufficient for practical purposes.

"I have now to consider the value of naphthalene.

"I am disposed to think that this body is of infinitely greater value than at first sight appears. Admitting that as an antiseptic it is inferior to the tar acids, nevertheless, so far as preservative action alone is concerned, it must not be supposed to be inoperative. Its special value, however, consists in helping to render the wood solid.

"But it may be said, granting this to be the case, naphthalene is so volatile, that the heat of the sun, especially the intense heat of an Indian climate, would soon drive the whole of it off. It is true that on exposing a block of creosoted timber in an oven to a temperature of 54·5° C., and this may be taken to be an extreme tropical heat, the door of the oven after a short time shows conclusively that some of the naphthalene in the sleeper has undergone volatilization by the heat applied.

"I would, however, direct attention to the following experiment:—

"I exposed a large block of creosoted timber (accurately weighed) to a temperature of 65·5° C. On weighing this at the end of twenty-four hours, I found it to have lost 1,200 grains. On exposing the same block to the same temperature for another twenty-four hours, it lost 135 grains; whilst on continuing the experiment for a third twenty-four hours, it lost only 15 grains. After this the loss was practically nil.

"I now planed off about a quarter of an inch of the block I had already heated. This done, I again exposed it to a heat of 54·5° C. for twenty-four hours, during which time it lost 1,150 grains. The loss on the second day was less than 100 grains, whilst on the succeeding days the loss was practically nil.

"The surface of the wood was again planed off, and similar experiments repeated a third time, with almost identical results.

"From numerous microscopical examinations of the timber, and from the experiments I have described, I consider that I am justified in drawing the following conclusions *re* naphthalene:—

"1st. That supposing, for the sake of argument, naphthalene possesses no great antiseptic power, nevertheless it acts beneficially by clogging up the pores of the wood, forming a more or less solid magma with the coagulated albumen. In this way it assists the physical part of the creosoting process, upon which the preservation of timber materially depends.

"2nd. That although a certain quantity of naphthalene would undoubtedly be volatilized by a tropical heat, nevertheless the loss would be practically limited to the *surface* of the timber, and would be complete a day or two after the exposure, the naphthalene in the deeper parts of the wood remaining fixed by incorporation with the albumen coagulated by the action of the tar acids.

"3rd. That inasmuch as the naphthalene cannot injure the action of the tar acids, or other constituents of the creosote, and is itself a positive benefit to the process, there is not only no object in requiring that the oil used for creosoting should be free from naphthalene, but that it would be unadvisable to demand such freedom.

"There are many other facts that in my judgment corroborate the views I have expressed. Thus I am given to understand that, during the twelve years after the process of creosoting was first introduced into India, the whole of the sleepers were prepared with heavy London creosote (that is, a creosote heavily charged with naphthalene), with the occasional admixture of a small quantity of Country oil for the purpose of dilution.

"It is perfectly certain, further, that it was on account of the good results so obtained that creosoting became a process of acknowledged utility.

"So far as I can learn it was not until the Country oils became more extensively used that any complaints respecting the inefficiency of the process arose. From independent inquiries, I think there is the strongest possible reason to believe that the sleepers that proved unsatisfactory had been prepared with Country and not with London oil.

"Nothing has impressed me more strongly in the course of these inquiries than the value of the heavy oils present in the creosote, that is, of the oils that do not distil over under 316° C. Of a certain antiseptic power, and very difficult of volatilization, they are, I believe, bodies of great value in the oil employed in the creosoting process."

Dr. Tidy's Specification for Creosote.

Taking into consideration the arguments of the report just quoted, Dr. Tidy recommended the following as a specification for creosote (amended in 1885):—

"(1) That the creosote shall be completely liquid at a temperature of 38° C., no deposit afterwards taking place until the oil registers a temperature of 35° C.

"(2) That the creosote shall contain at least 25 per cent. of constituents that do not distil over at a temperature of 316° C.

"(3) That tested by the process hereafter to be described, the creosote shall yield a total of 8 per cent. of tar acids.

"(4) That it shall contain no admixture of bone oil, shale oil, or of any substance not obtained from the distillation of coal tar, and that the first 25 per cent. of the distillate shall have a specific gravity greater than that of water.

Process to be adopted for determining the coal tar acids.

"(1) 100 cc. of the well mixed creosote are to be distilled at a temperature of 316° C. until no further distillate comes over. The distillate so obtained is to be mixed and well shaken in a stoppered flask with 30 cc. of a solution of caustic soda, sp. gr. 1·2 (5 E). The mixture is then to be heated. This done, the stopper is to be replaced in the flask, and the hot mixture again shaken vigorously for at least a minute.

"The contents of the flask are now to be poured into a separating funnel (Fig. 76), and the soda solution drawn off. The creosote is to be heated a second and a third time in a similar manner with the caustic soda solution, except that only 20 cc. of the soda solution shall be used for the second and third extraction, instead of 30 cc. as in the first extraction.

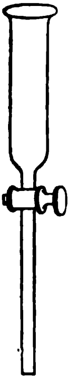

FIG. 76.

"(2) The three soda solutions are now to be mixed together. *When cold*, any particles of creosote are to be got rid of by means of a separating funnel. This done the solution to be thoroughly boiled, in order to expel the last traces of creosote present in the solution. The mixture is then allowed to be cooled. When cold 9 E H_2SO_4 is to be added (about 35 cc. will be required) until the solution becomes slightly acid to litmus. The whole is then to be poured into a separating funnel, and allowed to stand until perfectly cold and the tar acids well separated.

"(3) The tar acids are now to be dissolved in 20 cc. of the caustic soda solution, sp. gr. 1·2 (5 E), and 10 cc. of water. The mixture is then to be boiled and filtered through a funnel fitted with a plug of asbestos. The asbestos is to be washed with not more than 5 cc. of boiling water. The solution is to be allowed to cool *perfectly* in a 100 cc. measure. It is then to be rendered slightly acid, with 12 E H_2SO_4 (10 cc. will probably be found sufficient for this purpose). The whole is again allowed to stand for *two hours* until *perfectly cold*, when the percentage of the tar acids is to be read off.

Process to be adopted in estimating quantity of distillate.

"The operation is to be conducted in a retort (fitted with a thermometer), and heated with the naked flame of a Bunsen burner. The heat applied is to be gradually raised to 316° C., and continued at that temperature until no further matters distil over. The distillation of the 100 cc. should be completed within half an hour."

Sir F. Abel's Specification for Creosote.

The following is a specification for creosote which was drawn up by Sir Frederick Abel in January, 1884:—

"The creosoting liquor is to be of the description known as heavy oil of tar, obtained by the distillation of coal tar, and consisting of that portion of the distillate which comes over between the temperature of 176° C. and 405° C.

"The liquor must be free from admixture with any oil or other substance not obtainable from such distillate. It shall contain not less than 20 nor more than 30 per cent. of constituents that do not distil over at a temperature approaching 316° C.

"It must yield not less than 9 per cent. by volume of tar acids.

"The creosoting liquor must become completely fluid when raised to a temperature of 38° C., and exhibit no signs of any deposit on cooling down to a temperature of 32½° C.

"The specific gravity of the liquor must not be less than 1·035, and not more than 1·065, at a temperature of 32° C., as compared with water at 15½° C."

Creosote Oils.—The following table shows the character of various creosote oils :—

	Specific Gravity at 32° C.	Per cent. of Distillate below 315° C.	Per cent. of Tar Acids from Distillate.
A. Heavy London oils : highest	1·075	79	8·0
Ditto lowest	1·048	60	3·0
Average of 20 samples . .	1·0588	71·5	5·6
B. Partial runnings from London oils : highest . .	—	91	10·2
Ditto lowest . .	—	78	8·2
Average of 20 samples . .	—	82·8	91·5
C. English Country oils : highest	1·056	90	24·0
Ditto lowest	1·024	72	13·5
Average of 18 samples .	1·0335	81·8	18·6

Carbolineum.—This is a substance becoming much used as a timber preservative. It is, however, mostly used superficially for painting telegraph poles, wood pavements, fences, &c., and is said to effectually prevent water from getting into the wood. It has a remarkably high boiling point, and is prepared from the anthracene oil, squeezed out of rough anthracene, and then supposed to be mixed with sulphate of copper or chloride of zinc, and certain proportions of boiled linseed oil or resin. Owing to the absence of phenol, it may not be considered so effectual for the pickling of sleepers in the ordinary way as creosote would be.

The author has found that ordinary green oil obtained from coal tar is quite as effectual as a timber preservative as many samples of Carbolineum he has examined.

PART VIII

DISINFECTANTS.

The assay and valuation of the disinfectants treated of here, will include those that are, commercially speaking, the most in use, viz., carbolic acid, carbolic acid powders, chloride of lime (bleaching powder), and permanganate of soda or potash (Condy's Fluid).

Carbolic Acid (Phenol, C_6H_5HO).

Carbolic acid, which is a powerful antiseptic and disinfectant, is principally obtained from that fraction obtained on the distillation of coal tar which distils between 150° C. and 200° C. In the pure state it is a colourless crystallized solid, melting at 42·2° C., and boiling at 182° C. A commercial carbolic acid much used as a disinfectant is a dark-coloured liquid with a characteristic tarry smell, ranging in specific gravity from 1·04 to 1·06, and sometimes containing a considerable proportion of neutral tar oils of little use as a disinfectant.

Absolute crystallized phenol is miscible with water at the ordinary temperature, to the extent that the most concentrated solution contains about 73 per cent. of the pure acid, while the liquid thus produced is soluble in about 11·2 times its measure of water at 15° C. The solubility however, is greatly increased at higher temperatures, being miscible in all proportions with water at about 73° C. The pure acid is miscible in all proportions in benzol, ether, carbon disulphide, acetic acid (17 E), alcohol, and glycerine.

Carbolic acid acts as a powerful disinfectant and antiseptic owing to its property of coagulating albuminous substances, and preventing germinal life from propagating. A dilute solution of phenol is the most satisfactory agent we know of for arresting and preventing mortification.

By determining the neutral tar oils in a known volume of a sample of commercial carbolic acid, and subtracting the amount thus found from the volume of the original sample taken, an *approximate* idea of the value of a sample could thus be obtained.

Estimation of Neutral Tar Oils.—Measure out 5 cc. of the crude acid into a carbon tube, Fig. 9, add to this 10 cc. of a 2 E solution of sodic hydrate, and thoroughly shake for some time; the soda thus combines with the tar acids, forming phenate and cresylate of soda, &c., which is soluble in the aqueous solution. On allowing the mixture to stand some time, the neutral tar oils will settle out, sinking to the bottom or floating on the top, according to whether they are heavier or lighter than the aqueous liquid; 5 cc. of petroleum ether are now added, the mixture thoroughly shaken, the volume of the ethereal solution of the neutral oils is read off, and on subtracting the 5 cc. of added ether, the remainder is equal to the volume of neutral oils present in the 5 cc. of sample, which multiplied by 20 gives the percentage.

Estimation of Phenol and Cresol.—These are the principal acting disinfectants in commercial carbolic acid, and Dr. Tidy asserts that cresol (C_7H_7OH) is as equally efficacious as pure phenol (C_6H_5HO). If the amounts of these be required collectively, the volumetric process devised by Koppeschaar, depending upon the formation of tri-bromo-phenol ($C_6H_2Br_3OH$), and tri-bromo-cresol ($C_7H_4Br_3OH$) gives very fair results. The following are details of the process:—

Solutions required.—A solution of thiosulphate of soda

($Na_2S_2O_3$) of such strength that 1 cc. of it would be theoretically equivalent to ·005 grm. of iodine, is prepared by dissolving 9·764 grms. of crystallized thiosulphate of soda ($Na_2S_2O_3$ 5 H_2O) (previously dried between blotting paper) in distilled water, and diluting to 1,000 cc. at 15° C. It should be standardized by the method herein described with a solution of iodine containing 5 grms. per litre.

Starch Solution—Weigh out 1 grm. of starch, make into a thin paste with cold water, pour on 100 cc. of boiling water, stir, allow sediment to subside, and bottle the clear supernatant liquid.

Bromine Water.—Dissolve about 1½ grms. of liquid bromine in 1 litre of distilled water.

Potassium Iodide Solution.—Dissolve 125 grms. of pure KI in water, and dilute to 1,000 cc.

When all the above solutions have been made, dissolve 4 grms. of the sample in water, dilute to 1,000 cc. at 60° C. in a graduated stoppered flask, thoroughly mix, and put on one side while the bromine solution is valued as follows:—Fill a Mohr's burette with the standard thiosulphate solution, then measure off accurately 50 cc. of the bromine solution into a 100 cc. beaker and add 5 cc. of the potassic iodide. Iodine is thus liberated; now run in gradually the thiosulphate solution, stirring till the violet colour of the iodine has nearly all disappeared (the reaction here is:—$2\ Na_2S_2O_3 + 2\ I = 2\ NaI + Na_2S_4O_6$), then add 2 cc. of starch solution, when the blue colour of iodide of starch will be produced with the small quantity of free iodine still in solution; continue running in the thiosulphate solution until the last drop added completely decolourizes the solution. Note the volume required. 25 cc. of the solution of the sample (= 0·1 grm. original sample) are now poured into a graduated stoppered 500 cc. flask, and diluted quickly up to the mark with the bromine solution (= 475 cc.). Fix stopper, thoroughly shake for about ten minutes, and allow to stand for about half an hour, after which pour out into a litre beaker, containing 5 cc. of the iodide solution. Stir and titrate with the standard thiosulphate solution in the same manner as with the bromine solution, and note volume required.

From the following formula the percentage of carbolic and cresylic acids expressed as carbolic acid is obtained :—

Let X = thiosulphate required for 50 cc. bromine solution.
„ Y = „ „ for excess of bromine in the 25 cc. of the sample (= ·1 grm.).

Then $(9{\cdot}5\,x - y)\,0{\cdot}61753$ = percentage of carbolic acid.

Carbolic Acid Powders.

The best carbolic acid powders in the market are those which have for a base or absorbent, for the crude carbolic acid, substances which do not chemically combine with the carbolic acid, such as kaolin, calcium sulphate, kieselguhr, &c. Carbolic acid powders made with slaked lime appear to be of little use as disinfectants, since the carbolic acid thus combines with the lime, forming carbolate of lime. "Macdougall's Disinfecting Powder" contains a crude sulphite of lime as a base. "Calvert's Carbolic Acid Powder" is made by mixing the carbolic acid with the siliceous residuum left in the manufacture of alum from shale. There are five qualities made, containing 5, 10, 15, 20, and 50 per cent. of the crude acid.

Commercial carbolic acid powders may be considered of very fair quality if they contain from 15 to 20 per cent. of crude carbolic acid.

Method of Assay.—There are two methods in use for the assay of carbolic acid powders: (1) by distilling a known weight direct in a retort with the collection and examination of distillate, &c., a process applied to powders which contain the acid in a free state; and (2) liberation of combined acid with sulphuric acid and its subsequent extraction with benzol, &c.

(1) *Method for Powders containing Free Carbolic Acid.*—Weigh out 100 grms. of the averaged sample, transfer into a 150 cc. retort, attach a 100 cc. graduated tube as a condenser, apply a gentle heat at first to the retort with the naked

flame of a Bunsen burner, and gradually increase the heat until the contents of the retort are at a dull red heat, and no more liquid is being distilled. Note the volume distilled, and the amount of water separated out. It is important that the oily layer should be tested for neutral tar oils, which are apt to be used as a useless adulterant. Pour the liquid into a separator, separate the oily layer, and estimate the tar oils as described on page 344, and, if necessary, the carbolic acid by the Koppeschaar process.

(2) *Method for Powders containing Combined Carbolic Acid.*—This process can be applied with equal advantage to powders containing free carbolic acid without the addition of sulphuric acid. Weigh out 50 grms. of the sample into a 200 cc. glass mortar, mix with the aid of a pestle with 5 cc. of water, and then run in gradually 18 E sulphuric acid, stirring until a decided but slight excess is added; 50 cc. of water are now added, and the whole poured off into a capacious separator; 60 cc. of 90 per cent. benzol are now added, and the mixture thoroughly agitated for some time, to ensure the complete extraction of the crude carbolic acid by the benzol. It is then allowed to separate, the benzol solution distilled, and the residual crude carbolic acid thus isolated examined for tar oils and carbolic acid by the methods before mentioned.

Assay of Permanganate of Potash (*Condy's Fluid*).—The value of this useful disinfectant depends upon the amount of available oxygen present. The molecule of pure permanganate of potash ($K_2Mn_2O_8$) contains five atoms of available oxygen, capable of destroying by oxidation organic matter, germs of disease, &c., and in order to determine the amount of available oxygen present we must find out how much of a given substance capable of being oxidized by it to a definite form is required for a known weight. Pure crystallized ammonio-ferrous sulphate [$Fe(NH_4)_2(SO_4)_2\,6\,H_2O$] is a salt well adapted for this purpose, and an $\frac{E}{10}$ solution is of convenient strength to use, which is prepared by dissolving 39·2 grms. of the salt (previously dried between blotting paper) in recently boiled distilled water, and diluting to 1,000 cc. at 15° C.; 1 cc. of this solution corresponds to ·0008 grm. of available oxygen.

Five grms. of the sample are carefully weighed into a beaker, dissolved in recently boiled distilled water, diluted to 1,000 cc. at 15° C. in a graduated flask, thoroughly mixed, and any sediment present allowed to subside; 50 cc. of the clear solution (= ·25 grm. sample) are measured into a 150 cc. beaker, and 20 cc. of 5 E H_2SO_4 added. A burette is now filled with the ammonio-ferrous sulphate solution, and this is gradually run into the solution of the sample, stirring until the last drop added gives a slight but permanent pink coloration to the liquid, which can be best observed by holding a piece of tissue paper behind the beaker. Note the volume required. The reaction that takes place is as follows:—

$$10\ Fe(NH_4)_2(SO_4)_2 + 8\ H_2SO_4 + K_2Mn_2O_8 = 5\ Fe_2(SO_4)_3 + 2\ MnSO_4 + K_2SO_4 + 8\ H_2O + 10\ (NH_4)_2(SO_4).$$

The number of cc.'s of $\frac{E}{10}$ $K_2Mn_2O_8$ required × ·0008 × 4 × 100 = percentage of available oxygen present.

Assay of Chloride of Lime (*Bleaching Powder*).—This well known disinfectant is manufactured by passing chlorine gas over slaked lime *gradually*, so as not to raise the temperature of the substance, otherwise chlorate of lime will be produced, which is of no use as a disinfectant. The reaction which goes on when the temperature is kept low may be expressed as follows:—

$$3\ Ca(OH)_2 + 2\ Cl_2 = 2\ \underbrace{Ca\left\{\begin{matrix}OH\\OCl\end{matrix}\right. + CaCl_2 + 2\ H_2O}_{\text{Bleaching powder.}}$$

If the temperature be raised, it is decomposed with the formation of hydrate and chlorate of lime, and calcium chloride thus:—

$$3\ \underbrace{Ca_3H_6O_6Cl_4}_{\text{Bleaching powder.}} = 5\ CaCl_2 + \underbrace{Ca(ClO_3)_2}_{\text{Chlorate of lime.}} + 3\ CaH_2O_2 + 6\ H_2O.$$

The value of bleaching powder as a disinfectant, depends upon the liberation of hypochlorous acid, which is a very powerful oxidizing agent. The hypochlorous acid is brought about by the action of moist air containing carbonic acid, calcium carbonate being simultaneously produced. When used as a bleaching agent it is mixed with water; the article to be bleached is dipped into the mixture, and then into a dilute solution of sulphuric acid; chlorine is thus liberated in a nascent state in the fibre of the material, which combines with the hydrogen of water, thus liberating nascent oxygen, which in its turn destroys the colouring matter of the material. Good bleaching powder should contain about 35 per cent. of available chlorine, the maximum amount that can be present being about 39 per cent. But owing to careless manufacture, exposure to air, &c., the amount of available chlorine may be anything below this amount.

The best method for determining the amount of available chlorine in bleaching powder is that devised by Penot, which depends upon the oxidation of arsenious acid (As_2O_3) into arsenic acid (As_2O_5) by the sample, thus:—

$$As_2O_3 + Ca(ClO)_2 = As_2O_5 + CaCl_2.$$

Solutions Required.—Deci-Normal Sodic Arsenite.—Weigh out accurately 4·95 grms. of pure arsenious acid into a 300 cc. beaker, together with 25 grms. of pure crystallized carbonate of soda; pour on to the mixture 250 cc. of hot water, and heat and stir until all goes into solution; cool, pour into a graduated stoppered litre flask, dilute to 1,000 cc. at 15° C., and thoroughly mix. 1 cc. of the $\frac{E}{10}$ solution = ·00355 grm. chlorine.

Starch Test Papers.—Weigh out about 3 grms. of starch into a glass mortar, and make into a paste with about 50 cc. of water, with the aid of a pestle. Pour into a beaker and gradually add 200 cc. of boiling water, stirring, and then add 1 grm. of carbonate of soda and 1 grm. of potassic iodide dissolved in a little water, and dilute the whole to about 500 cc.

Strips of filter paper are moistened with this solution, and after carefully drying they are put into a stoppered bottle ready for use.

The Analytical Process. — 10 grms. of the well mixed sample are weighed into a 150 cc. glass mortar, 60 cc. of water poured on it, the mixture thoroughly lixiviated with the aid of a pestle, and the sediment allowed to subside as far as possible; after which the supernatant and turbid solution is decanted off into a graduated stoppered litre flask; more water is added to the residue in the mortar and mixed as before, and the liquid poured off into the flask; the lixiviation with portions of water is repeated until the whole of the sample is thus got into the flask, and then finally diluted to 1,000 cc. at 15° C.

Accurately measure off 50 cc. of the solution (= ·5 grm. original sample) into 200 cc. beaker, and gradually run in the standard $\frac{E}{10}$ sodic arsenite solution from a burette, stirring until a drop of the solution, taken out with the stirrer, no longer produces a blue stain of iodide of starch upon one of the prepared starch papers.

Example.—10 grms. of a sample of bleaching powder was treated as above, and 50 cc. (= ·5 grm. sample) of the solution required 48·5 cc. of the arsenious solution to produce no further blue coloration of the starch paper.

1 cc. of $\frac{E}{10}$ arsenious solution = ·00355 grm. chlorine.

48·5 × ·00355 = ·1722 grm. chlorine in 0·5 grm. of sample.
·1722 × 2 × 100 = 34·44 per cent. available chlorine.

PART IX.

EXPLOSIVES.

Nitro-Glycerine [$C_3H_5(NO_3)_3$].—Nitro-glycerine is prepared on a large scale by first mixing four parts of 36 E H_2SO_4, and one part of 22 E HNO_3 (fuming) in a wooden vessel lined with lead, and allowing to cool, after which one part of glycerine is allowed to run into the mixture in a thin stream. The whole is agitated with compressed air and kept cool with cold water, which runs through leaden worms contained in the nitrating vessel. The temperature of nitration should not exceed 25° C. On allowing to stand for some time, the nitro-glycerine separates as an oily liquid on the surface of the acid, which is separated, washed first with water, and finally with a dilute solution of sodic carbonate to eliminate the last traces of acids. The sulphuric acid takes no part in the reaction beyond keeping the nitric acid up to the proper strength by combining with the water produced in the process. The reaction may be represented as follows :—

$$C_3H_5(OH)_3 + 3\ HNO_3 = C_3H_5(ONO_2)_3 + 3\ H_2O.$$

It is important that all free acid should be perfectly neutralized, since, if any is left in the finished product, it is liable to spontaneously decompose. The specific gravity of nitroglycerine is 1·600 at 15° C., and it solidifies at 8° C. When heated to about 257° C., it decomposes with a sharp detonation; a similar result is obtained when a little of the substance is smartly struck or compressed. When ignited in air it burns with a greenish flame without explosion. When completely exploded the products of combustion are carbonic acid, nitrogen, water, and free oxygen which may be represented by the equation:—

$$2\ C_3H_5\ (NO_3)_3 = 6\ CO_2 + 5\ H_2O + N_6 + O.$$

According to Bloxam, when nitro-glycerine is dissolved in alcohol and warmed with ammonium sulphide it is decomposed, sulphur being separated, the reaction being:—

$$C_3H_5(ONO_2)_3 + 3\ NH_4HS = C_3H_5(OH)_3 + 3\ NH_4NO_2 + S_3.$$

The value of nitro-glycerine can be determined from the amount of nitric oxide liberated when a weighed portion is treated with sulphuric acid over mercury in a nitrometer, as in the determination of nitrates in waters, page 206. This process was devised by Hempel and Lunge, who proceed as follows: 0·2 grm. is introduced into the cup of the nitrometer previously filled with mercury, and 2 cc. of 36 E H_2SO_4 added, stirred with a glass rod, and the mixture made to run into the nitrometer. The cup of the nitrometer is rinsed out with successive small portions of 36 E H_2SO_4, 3 cc. in all being used. The mixture is then thoroughly shaken up with the mercury until no further evolution of nitric oxide occurs. The number of cc's. of nitric oxide thus produced corrected to 0° C., and 760 mm. mercury, multiplied by $\frac{3·39 \times 100}{200}$ gives the percentage of nitro-glycerine present.

The Glycerine used.—It is very essential that the quality of the glycerine used in the manufacture of nitro-glycerine should be of the best. The minimum specific gravity should be 1·261 at 15° C., and must be free from lime, iron, alumina, chlorides and fatty acids. Not more than 0·25 per cent. of inorganic and organic matter should be left when 25 grams of the sample are evaporated in a weighed platinum dish and the residue dried in an air bath at 160° C., until the weight remains constant. The residue may be incinerated and the amount of inorganic matter present thus estimated.

A good test of the quality of glycerine is to measure the proportion of nitro-glycerine yield on nitration. Theoretically, 1 lb. of glycerine should yield 2·47 lbs. of nitro-glycerine, but on the manufacturing scale only about 2 lbs. are produced, due to unavoidable waste and the formation of other nitro-compounds which are carried away with the washings.

In a laboratory experiment 100 grams of good glycerine should yield 210 grams of nitro-glycerine. The following is a method of testing the yield of the nitro-compound :—Pour 80 cc. of mixed acids (3 of 16 E HNO_3 to 5 of 36 E H_2SO_4 by weight) into a 300 cc. beaker, and its contents into a bowl of cold water, then run in gradually, stirring, 10 grams of the glycerine contained in a small beaker; rinse the beaker out with a little of the mixed acids and stir. The whole is now poured into a 100 cc. graduated burette, and the mixture allowed to settle for about half an hour or so, when all the nitro-glycerine will have separated, the volume separated is noted and its quantity by weight calculated from the specific gravity = 1·60.

The following is an example of a determination :—

10 grams of glycerine gave 13·2 cc. of nitro-glycerine.

Then, $13{\cdot}2 \times 1{\cdot}6 \times 10 = 211{\cdot}2$ grams per 100 grams of sample.

Dynamite.—Dynamite is usually a plastic body varying in colour from brown to reddish brown, and is prepared by mixing nitro-glycerine with a highly porous substance such as kieselguhr, largely composed of silica.

Dynamite is sent out in cylindrical rolls of a thickness suitable to the size of bore holes required, and covered with parchment paper so as to form cartridges. Packets of five pounds of cartridges are made, and ten of these packages go to a box.

Explosives of the dynamite class become hard or frozen when exposed to cold, about 40° Fahr. being the usual freezing point, but they still remain frozen when warmed considerably above this point. In this condition dynamite is of no use for blasting purposes unless first thawed. Frozen dynamite is much less sensitive to a blow or detonation than it is in the unfrozen state, and on ignition it is very liable to explode, which is not the case with unfrozen dynamite, the latter burning away without explosion. Great care should be exercised in the thawing of frozen dynamite cartridges, since there have been a great number of fatal accidents brought about by warming the cartridges before a fire, &c., and by methods of which no gauge of temperature can be obtained. It will appear obvious, when it is considered how explosive nitro-glycerine compounds become when they are heated near their points of explosion, viz. 360° Fahr., that such methods of thawing are highly dangerous.

A safe method of thawing is to employ an apparatus of the glue-pot principle. Hot water at a temperature of about 140° Fahr. is poured into the outer vessel, then the dry inner pot containing the cartridges is allowed to digest in it until the latter has attained its normal condition.

There are two varieties of dynamite licensed in this country, known as Dynamite No. 1 and Dynamite No. 2, the latter being a slower and milder variety than the former. The following are their legal definitions :—

"*Dynamite No.* 1, consisting of not more than 75 parts by weight of thoroughly purified nitro-glycerine, uniformly with 25 parts by weight of—

"(*a*) An infusorial earth known as *kieselguhr*, or

"(*b*) A non-explosive mixture of kieselguhr with such other ingredients and in such proportions as may for the time being be sanctioned by the Secretary of State.

Provided—

"(1) That the said *kieselguhr*, or (*b*) non-explosive mixture, shall be sufficiently absorbent in quality when mixed in the above proportions to prevent exudation of nitro-glycerine; and,

"(2) That there may be added to the kieselguhr or non-explosive mixture an amount of carbonate of ammonium not exceeding 1½ parts by weight in every 100 parts by weight of finished dynamite."

The ingredients at present sanctioned under (*b*) are :—

Carbonate of sodium . .	In all 8 parts (or less) by weight, in substitution for an equal amount by weight of Kieselguhr.
Sulphate of barium . . .	
Mica	
Talc	
Ochre	

Provided that the total amount of carbonate of sodium shall in no case exceed 3 parts by weight in every 100 parts by weight of the finished dynamite.

"*Dynamite No.* 2, consisting of not more than 18 parts by weight of thoroughly purified nitro-glycerine, uniformly mixed with 82 parts by weight of a pulverised preparation, composed of nitrate of potassium 71 parts, charcoal not less than 10 parts, and purified paraffin (*or* ozokerit) 1 part (*or* nitrate of potassium 72 parts, and charcoal not less than 10 parts) by weight, and sufficiently absorbent in quality when mixed in the above proportions to prevent exudation of nitro-glycerine."

There are numerous explosives of the dynamite class now in the market, containing a variety of substances, many of which are not classified among the explosives licensed in this country. For the composition of various kinds, see page 361.

Determination of Water.—Water cannot be determined by drying a weighed portion of the sample in a water oven, since nitro-glycerine is thereby volatilized. Two grms. of the sample are reduced carefully, as finely as possible with the aid of a horn spatula, and dried in a vacuum over strong sulphuric acid until the moisture is eliminated.

Determination of Nitro-Glycerine.—When other substances soluble in methylated ether are absent, the nitro-glycerine can be determined by exhausting 2 grms. of the sample with methylated ether in a Soxlet's fat extraction tube (Fig. 77), and weighing the residuum, the loss being taken as nitro-glycerine.

The nitro-glycerine cannot be accurately determined by the evaporation of the ethereal extract and weighing the residue, since there is a sensible loss of nitro-glycerine by this means.

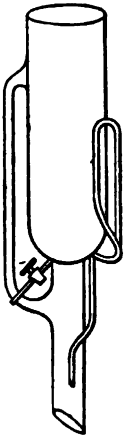

FIG. 77.

Gelatine Dynamite, Gelignite, or Blasting Gelatine.—The writer is indebted to Mr. P. G. Sanford for a private communication on the methods of analysis.

1. Weigh out about 10 grams and place over $CaCl_2$, in a vacuum; after a few days reweigh, and again place in exicator until constant in weight. Loss = moisture.

2. Place the dry sample in a 500 cc. Erlenmeyer flask, and pour over it 250 cc. of ether-alcohol (2 : 1), shake, and allow to stand over-night; when all the nitro-glycerine and collodion cotton has dissolved, filter through a tarred filter, and wash with ether-alcohol.

3. *Residue*, which consists of KNO_3 and wood pulp, and any sodic carbonate which may be present, is dried at 60° C. and weighed; the residue is then detached from the filter and boiled several times in distilled water, decanting the water each time through a weighed filter. The residue upon the filter must be well washed with hot water dried at 100° C. and weighed = *wood pulp*. The solution which contains the KNO_3 is evaporated to dryness in a platinum dish and weighed = KNO_3. This residue must be tested for the presence of sodic carbonate.

4. *Solution*—contains the collodion cotton and nitro-glycerine; to this solution add excess of chloroform, which precipitates the nitro-cotton; filter through a linen filter (this is best done by keeping the funnel warm), dry at 40° C. in an air-bath; when dry, detach from filter, and redissolve ether-alcohol, and then again precipitate with chloroform, filter, &c., and dry at between 40° and 90° C.; detach from filter and weigh upon a watch-glass = *nitro-cotton*.

The solution may be evaporated to obtain the nitro-glycerine, but the result will be much too low and the nitro-glycerine is best found by difference.

The following are analyses of various nitro-compounds analysed by the foregoing methods:—

	Gelatine dynamite.	Gelignite.	Blasting gelatine.
Nitro-cotton	= 7·632 %	4·888 %	8·900
Nitro-glycerine	= 71·128 „	60·514 „	91·100
Wood pulp	= 4·259 „	7·178 „	—
Potassic nitrate	= 16·720 „	27·420 „	—
Water	= 0·261 „	Nil. „	—
	100·000	100·000	100·000

ANALYSIS OF KIESELGUHR DYNAMITE.

Nitro-glycerine = 73·431
Kieselguhr = 26·370
Water = 0·199
100·000

COLLODION COTTON.

Collodion cotton = 98·85 }
Trinitro-cellulose, or gun-cotton = 1·15 } = Nitrogen = 11·49 %
Non-nitrated cellulose = trace
100·000

The following is a tabulated scheme, arranged by Allen, for the analysis of nitro-glycerine compounds:—

SCHEME FOR ANALYSING NITRO-GLYCERINE COMPOUNDS (ALLEN).

<table>
<tr><td colspan="8">Exhaust the previously dried sample with anhydrous ether in a fat extraction apparatus.</td></tr>
<tr><td colspan="3"><i>Solution</i> :—Divide into two equal parts.</td><td colspan="5"><i>Residue</i> :—Dry, weigh, and exhaust with water in a Soxlet tube.</td></tr>
<tr><td rowspan="4">A.—Allow the ether to evaporate at ordinary temperature, and dry the residue in vacuo over 36 E H_2SO_4 and weigh. Weight gives the sum of nitro-glycerine, resin, camphor (sulphur), and paraffin. Determine the nitro-glycerine by method given.</td><td colspan="2" rowspan="2">B.—Add a few drops of phenolphthalein and titrate with standard E alcoholic potash. 1 cc. of E KHO = 0·330 grm. resin. Add 10 cc. more E KHO and evaporate. Dissolve residue in water, shake with ether, and separate.</td><td rowspan="4"><i>Solution</i> :—Contains metallic nitrates, chlorates, soluble carbonates, &c., the sum of which (exclusive of ammonium carbonate) can be determined by evaporating the solution to dryness at 100° C., and weighing the residue. The nitrates can be here determined by the nitrometer.</td><td colspan="4"><i>Residue</i> :—Dry, weigh, and agitate an aliquot part with 36 E H_2SO_4 over mercury in nitrometer. NO formed is from cellulose nitrates. If any NO is evolved treat remainder of residue with a mixture of two volumes of ether to one of absolute alcohol.</td></tr>
<tr><td rowspan="3"><i>Solution</i> :—Evaporate and weigh residue, consisting of mono- and di-nitro-cellulose, collodion-cotton, which may be further examined in the nitrometer.</td><td colspan="3"><i>Residue</i> :—Dry, weigh, and treat a weighed portion in nitrometer, calculated evolved gas to trinitro-cellulose, $C_6H_7(NO_2)_3O_5$. If present exhaust the remainder with acetic ether.</td></tr>
<tr><td rowspan="2"><i>Ethereal solution</i> evaporated leaves the paraffin.</td><td rowspan="2"><i>Aqueous liquid</i> :—Add bromine, acidify with 10 E HCl, separate any resin, and precipitate filtrate with E $BaCl_2$. $BaSO_4 \times ·1373 =$ sulphur.</td><td rowspan="2"><i>Solution</i> contains trinitro-cellulose.</td><td colspan="2"><i>Residue</i> :—Weigh, ignite, and reweigh.</td></tr>
<tr><td><i>Loss</i> equals cellulose, sawdust, charcoal, coal, &c.</td><td><i>Residue</i> consists of kieselguhr, chalk, and other mineral substances.</td></tr>
</table>

Gunpowder.—Gunpowder is a mechanical mixture of nitre, charcoal and sulphur. Good charcoal obtained from dogwood, willow, or alder, is the best for the purpose; the sulphur preferred is that which has been distilled and ground to a fine meal; and nitre only of the first quality is employed.

The proportion of these ingredients varies in different countries, and also according to the purpose for which the gunpowder is proposed to be used.

The following table shows the percentage compositions of gunpowders of various countries:—

	Nitre.	Sulphur.	Charcoal.
English and Austrian (musket) .	75	10	15
Prussian (musket)	75	11·5	13·5
Swedish ,, ...	75	9	16
Chinese	75·7	9·9	14·4
French (musket)	75·0	12·5	12·5
,, (sporting)	76·9	9·6	13·5
,, (blasting)	62	20	18

The products of combustion of gunpowder are very complicated, the composition varying according to the manner in which the powder is fired. Karolyi, Noble and Abel succeeded in making an analysis of the products of combustion of gunpowder.

The following shows some of the results obtained by firing three kinds of powder under the conditions of artillery practice:—

(1) COMPOSITION OF THE POWDER USED.

		Ordnance powder.		Small arms powder.		Pebble powder (Noble and Abel).	
Nitre . . .		73·78		77·15		74·67	
Sulphur . .		12·80		8·63		10·07	
Charcoal.	Carbon . .	10·88	13·39	11·78	14·27	12·12	14·22
	Hydrogen .	0·38		0·42		0·42	
	Oxygen . .	1·82		1·79		1·45	
	Ash . .	0·31		0·28		0·23	
Water . . .		—		—		0·95	
		99·97		100·05		99·91	

(2) Products of Combustion of 100 Parts by Weight.

	Ordnance powder.	Small arms powder.	Pebble powder (Noble and Abel).
Nitrogen	9·77	10·06	11·39
Carbonic acid	17·39	21·79	27·70
Carbonic oxide	2·64	1·47	4·73
Hydrogen	0·11	0·14	0·05
Sulph. hydrogen	0·27	0·23	0·84
Marsh gas	0·40	0·49	0·12
Total gaseous	30·58	34·18	44·83
Ammonic sesquicarbonate	2·68	2·66	0·04
Potassic sulphate	36·95	36·17	6·58
„ carbonate	19·40	20·78	30·98
„ thiosulphate	2·85	1·77	3·38
„ sulphide	0·11	0·00	10·55
Charcoal	2·57	2·60	—
Sulphur	4·69	1·16	3·40
Loss	0·17	0·68	—
Potassic sulphocyanate	—	—	0·13
„ nitrate	—	—	0·11
Total solid	69·42	65·82	55·17

(3) Products of Combustion by Volume in 100 of Gas.

	Ordnance powder.	Small arms powder.	Pebble powder (Noble and Abel)
Nitrogen	37·58	35·33	32·19
Carbonic acid	42·74	48·90	49·82
Carbonic oxide	10·19	5·18	13·36
Hydrogen	5·93	6·90	2·08
Sulphuretted hydrogen	0·86	0·67	1·96
Marsh gas	2·70	3·02	0·58
	100·00	100·00	99·99

The Analysis of Gunpowder.—*Estimation of Moisture.*—Weigh out 5 grms. of the powdered sample into a porcelain basin, and dry in *vacuo* over sulphuric acid until no further loss in weight occurs. The loss = moisture.

Estimation of Nitrogen.—The residue from the estimation of moisture is exhausted with water, and filtered through a tube containing a plug of glass wool, as in Fig. 5, into a weighed dish. When all soluble matter has been extracted, the solution is evaporated to dryness and the residue weighed. The residue should be again dissolved in water, and tested for sulphates and chlorides, the sulphates being precipitated with nitrate of barium, and the resulting $BaSO_4$ filtered off and estimated if necessary. The chlorides could be determined in the filtrate by precipitating with nitrate of silver, and filtering the resulting AgCl.

Estimation of Sulphur.—The residue left in the tube after the exhaustion with water, is dried in the tube by passing a current of air through, which has been previously dried by passing through sulphuric acid. The sulphur is then estimated in the manner described for the estimation of sulphur in spent oxide, page 331.

The Charcoal may be taken by difference. If it be necessary to determine the quality of the charcoal, this could be obtained by the method given on page 129 for the analysis of coal.

Patent Explosives.—Subjoined will be found the composition of a few of the many patent explosives now in existence*:—

CLASS I. NITRATED POWDERS.

Bolton's Powder.

	Parts.
Carbonate of copper	8
Graphite	10
Prepared quicklime	14
" alum	50
" sugar	350
Nitrate of soda	350
Soda ash	20
Ferrocyanide of potassium	300
Charcoal	30
Carbonate of potash	450

Pyronome.

	Parts.
Nitrate of soda	52·5
Spent tan	27·5
Sulphur	20·0

Diorrexin (*an Austrian explosive*)

Nitrates of potash and soda	60
Sulphur	12
Sawdust	10
Charcoal	7
Picric acid	1·5
Moisture	7·5

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* For further information with regard to the preparation, composition, &c., of various explosives, *see* Major Cundill's "Dictionary of Explosives" and Mr. Eissler's "Handbook of Modern Explosives."

CLASS I. NITRATED POWDERS (*continued*).

Johnite.

	Parts.
Nitrate of potash	75
Sulphur	10
Lignite	10
Picrate of soda	3
Chlorate of potash	2

Pudrolithe.

Saltpetre	68
Sulphur	12
Charcoal	6
Nitrate of baryta	3
,, ,, soda	3
Sawdust	5
Spent tan	3

Courteille's Powder.

Nitrate of soda or potash	60 to 75
Sulphur	10 ,, 12
Charcoal	7 ,, 10
Peat and hard coal	9 ,, 12
Combined metallic sulphates	2 ,, 4
Oleaginous matter, animal or vegetable (or tar), refined or crude	1 ,, 3

Davey's Powder.

	Parts.
Saltpetre	64
Sulphur	16
Charcoal	12
Flour, bran, or starch	8

Pyrolithe.

Saltpetre	51·5
Nitrate of soda	16·0
Sulphur	20·0
Sawdust	11·0
Charcoal	1·5

Safety Blasting Powder.

Saltpetre	70
Sulphur	12
Lampblack	5
Spent tan or sawdust	13
Sulphate of iron	2

Schäffer's Powder.

Saltpetre	30 to 38
Nitrate of soda	40
Sulphur	8 to 12
Charcoal	7 ,, 8
Rochelle salt	4 ,, 6

Violette's Powder.

Nitrate of soda	62·5
Acetate of soda	37·5

CLASS II. CHLORATE POWDERS.

Dynamogen.

	Parts.
Yellow prussiate of potash	17
Water	150
Charcoal	17

After boiling, and cooled, there are added :—

Potash	35
Chlorate of potash	70
Starch	10
Water	50

Jaline.

	Parts.
Nitrate of potash	65 to 75
Sulphur	10
Lignite	10 to 50
Picrate of soda	3 ,, 8
Chlorate of potash	2

Knaffl's Powder.

Chlorate of potash	46
Nitrate of potash	26
Sulphur	15
Ulmate of ammonia	10

CLASS II. CHLORATE POWDERS (*continued*).

Ehrhardt's Powders.

	Artillery. Pts.	Blasting. Pts.	Shells Pts.
Chlorate of potash	1	1	1
Saltpetre	1	1	—
Charcoal	—	4	—
Tannic acid	1	2	1

Goetz' Powder.

	Parts.
Chlorate of potash	10
Glucose solution	10
Charcoal in powder	3
Sulphur	2
Amorphous phosphorus	1
Picrate of lead	3

Nisser's Powder.

	Parts.
Yellow prussiate of potash	1·5
Bichromate of potash	2·0
Chlorate of potash	10·5
Nitrates of soda and potash	44·5
Vegetable matter	6·5
Mineral and vegetable carbon	19·5
Sulphur	15·5

Pyronome.

Saltpetre	69
Sulphur	9
Charcoal	10
Antimony	8
Chlorate of potash	5
Rye flour	4

CLASS III. DYNAMITE POWDERS.

Atlas Powders.

	A. Pts.	B. Pts.
Sodium nitrate	2	34
Wood fibre	21	14
Magnesium carbonate	2	2
Nitroglycerine	75	50

Castellanos Powder.

	Parts
Nitroglycerine	40
Nitrate of Potash	25
Picrate	10
Sulphur	5
Insoluble salts (silicates, &c.)	10
Carbon	10

Petralithe.

Nitroglycerine	640
Nitrate of ammonia, soda or urea	120
Palmitinate of cetyl	2½
Carbonate of lime	2½
Animal or vegetable charcoal	230
Bicarbonate of soda	5

Vulcan Powder.

	Parts.
Nitroglycerine	30
Nitrate of soda	52·5
Sulphur	7
Charcoal	10·5

Diaspon.

Nitroglycerine	47	to	63
Nitrocotton	0·5	„	3·0
Nitrate of soda	22	„	23
Wood cellulose	8	„	18
Sulphur	3	„	9

Dualine.

Nitroglycerine	50
Fine sawdust	30
Saltpetre	20

Hercules Powders.

	No. 1. Pts.	No. 2. Pts.
Carbonate of magnesia	20·85	10·00
Nitrate of potash	2·10	31·00
Chlorate of potash	1·05	3·34
White Sugar	1·00	15·66
Nitroglycerine	75·00	40.00

Class III. Dynamite Powders (*continued*).

Rendrock.

	Parts.
Nitrate of potash	40
Nitroglycerine	40
Wood fibre	13
Paraffin or pitch	7

Vigorite.

	Parts.
Nitroglycerine	30
Nitrate of soda	60
Charcoal	5
Sawdust	5

Class IV. Guncotton Class.

Ammonia Nitrate Powder.

	Parts
Ammonium nitrate	80
Potassium chlorate	5
Nitroglucose	10
Coal tar	5

Nitronaphthalene.

Nitronaphthalene	10
Saltpetre	75
Charcoal	12·5
Sulphur	12·5

Petrofacteur.

Nitro-benzene	10
Chlorate of potash	67
Nitrate of potash	20
Sulphide of antimony	3

Johnson's Powders.

	For Military Arms. Parts.
Nitrocellulose	50
Potassium nitrate	40
Torrefied starch	10

Bellite, Securite, Roburite.

These explosives, which have advantages for firing in coal mines, are mixtures of nitrate of ammonium with di or tri nitro-benzole, or chlorinated di nitro-benzole.

Schultze Powder.

	Parts.
Soluble nitrolignin	24·83
Insoluble nitrolignin	23·36
Lignin	13·14
Nitrate of potash and barium	32·35
Paraffin	3·65
Matters soluble in alcohol	0·11
Moisture	2·56

Class V. Picric Powders.

Designolles Powders.

	For Torpedoes and Shells. Pts.	Small Arms Pts.
Picrate of potash	55	28·6
Saltpetre	45	65·0
Charcoal	—	6·4

Victorite.

	Parts.
Chlorate of potash	80
Picric acid	110
Nitrate of potash, soda, or baryta	10
Charcoal	5

Melinite and Lyddite.

Picric acid embedded in collodion. Also fused picric acid used with a gun-cotton primer.

Picric Powder.

(ABEL'S.)

	Parts.
Saltpetre	3
Picrate of Ammonia	2

Turpin's Explosives.

Same as Melinite.

Emmensite.

This explosive is produced by treating picric acid with fuming nitric acid. The crystals thus produced are mixed with a nitrate.

Tschirner's Powder.

	Parts.
Picric Acid	57
Chlorate of Potash	43
Resin	5

This mixture is moistened with benzene or petroleum to dissolve the resin.

Bronolithe.

	Per cent.
Barium sodium picrate	15 to 30
Lead „ „	8 „ 30
Potassium „ „	2 „ 10
Nitronaphthalene	5 „ 20
Saltpetre	20 „ 40
Sugar	1·5 „ 3
Gum	2 „ 3
Lampblack	0·5 „ 4

DETERMINATION OF STABILITY OF EXPLOSIVES.

Instructions as to Tests issued by the Home Office.

Heat Test as applied to Explosives of the Nitro-Compound Class (as defined by Order in Council No. 1, of 5th August, 1875).

GENERAL INSTRUCTIONS.

Apparatus required.

(1) A water bath, consisting of a spherical glass or copper vessel (*a*), Fig. 78, of about 8 inches diameter, and with an aperture of about 5 inches; the bath is filled with water to within a ½ inch of the edge. It has a loose cover of sheet

copper about 6 inches in diameter (*b*), and rests on a tripod stand about 14 inches high (*c*), which is covered with coarse iron wire gauze (*e*), and is surrounded with a screen of thin sheet tin or copper (*d*). Within the latter is placed an argand burner (*f*) with glass chimney. The cover (*b*) has four holes arranged as seen in Fig. 79, No. 4 to receive the regulator,

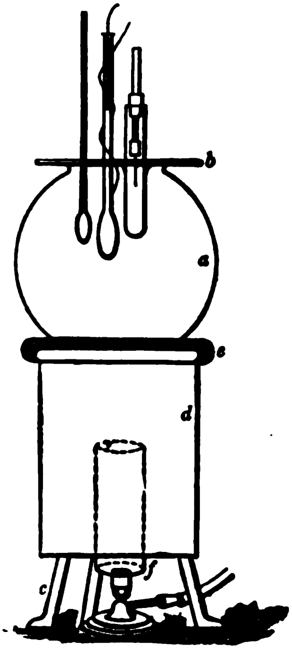

FIG. 78.

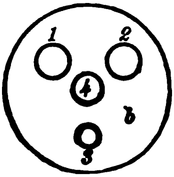

FIG. 79.

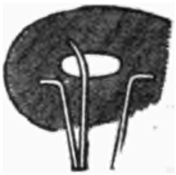

FIG. 80.

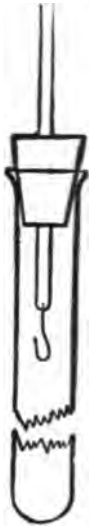

FIG. 81.

No. 3 the thermometer, Nos. 1 and 2 the test tubes containing the gun-cotton or other materials to be tested. Around holes 1 and 2 on the other side of the cover are soldered three pieces of brass wire with points slightly converging (Fig. 80); these act as springs and allow the test-tubes to be easily placed in position and removed.

*(2) Scheibler's or Page's temperature regulator.

*(3) Two cells of Le Clanché's battery No. 1 } if Scheibler's regulator is used.
*(4) A few yards of insulated copper wire }

(5) Test tubes from 5¼ to 5½ inches long, and of such diameter that they will hold from 20 to 22 cubic centimetres of water when filled to a height of 5 inches.

(6) Indiarubber stoppers, fitting the test tubes and carrying an arrangement for holding the test paper, viz., a narrow glass tube passing through the centre of the stopper, drawn out so as to form a hook, or terminating in a platinum wire hook (Fig. 81).

(7) A thermometer, with range not less than from 30° to 212° Fahrenheit.

(8) A minute clock.

Materials Required.

(*a*) *Test Paper.*—The test paper is prepared as follows:—45 grains of white maize starch (corn-flour), previously washed with cold water, are added to 8¾ ounces of distilled water; the mixture is stirred, heated to boiling, and kept gently boiling for ten minutes; 15 grains of pure potassium iodide (*i.e.*, which has been re-crystallised from alcohol) are dissolved in 8¾ ounces of distilled water. The two solutions are thoroughly mixed and allowed to get cold. Strips or sheets of white English filter paper, previously washed with water and re-dried, are dipped into the solution thus prepared, and allowed to remain in it for not less than ten seconds; they are then allowed to drain and dry in a place free from laboratory fumes and dust. The upper and lower margins of the strips or sheets are cut off, and the paper is preserved in well-stoppered or corked bottles *and in the dark.* When the paper is freshly prepared, and as long as it remains in good condition, a drop of dilute acetic acid, put on the paper with a glass rod, produces no coloration.

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* This is not absolutely required, as the temperature of the bath can be kept constant by proper attention to the heating flame.

In process of time, however, the stronger the light to which the paper is exposed, the sooner a drop of the acid produces a brown or bluish coloration (a single hour of direct sunlight produces a marked effect), and whenever this is the case the paper should be rejected. On this account it is advisable not to prepare too much of the test paper at one time, but to prepare it fresh every month or so.

(*b*) *Standard Tint Paper*.—A solution of caramel in water is made of such concentration that when diluted one hundred times (10 c.c. made up to 1 litre) the tint of this diluted solution equals the tint produced by the Nessler test in 100 c.c. water containing 0·000075 grm. of ammonia or 0·00023505 grm. of chloride of ammonium. With this caramel solution lines are drawn on strips of white filter paper (which paper must be carefully washed with distilled water in the first instance, to remove any traces of bleaching matter, and dried) by means of a clean quill pen. When the marks thus produced are dry, the paper is cut into pieces of the same size as the test paper previously described, in such a way that each piece has a brown line across it near the middle of its length, and only such strips are preserved in which the brown line has a breadth varying from ½ mm. to 1 mm. ($\frac{1}{50}$ inch to $\frac{1}{25}$ inch).

Testing Dynamite, Blasting Gelatine, and other Explosives of the First Division of the Nitro-compound Class.

A.—DYNAMITE, &c., &c.

Nitro-glycerine preparations, from which the nitro-glycerine can be extracted in the manner described below, *must* satisfy the following test, otherwise they will not be considered as manufactured with "thoroughly purified nitro-glycerine" within the terms of the licence.

This test, however, though at present looked upon as the most important, as far as testing the purity of the nitro-glycerine is concerned, is only one of several which any given sample of nitro-glycerine preparation has to satisfy in order to establish its compliance with the definition in the licence.

The test, although at present accepted as regulating and defining the meaning of the terms "thoroughly purified," may nevertheless be modified or superseded if, in the opinion of the Home Office, such alteration may at any time be deemed necessary.

Apparatus Required.—A funnel 2 inches across (*d*), a cylindrical measure divided into grains (*e*), see Fig. 82.

Mode of Operation.—About 300 to 400 grains of dynamite (*b*) finely divided are placed into the funnel, which has previously been loosely plugged by some freshly-ignited asbestos (*a*).

The surface is smoothed by means of a flat-headed glass rod or stopper, and some clean washed and dried kieselguhr (*c*) is spread over it to the depth of about ⅛ inch.

Water is next carefully dropped from a wash bottle upon this kieselguhr, and when the first portion has been soaked up, more is added; this is repeated until sufficient nitro-glycerine has been collected in the graduated measure (*e*) below.

If any water should have passed through with the nitro-glycerine, it should be removed with a piece of blotting paper, and the nitro-glycerine, if necessary, filtered through a dry paper filter.

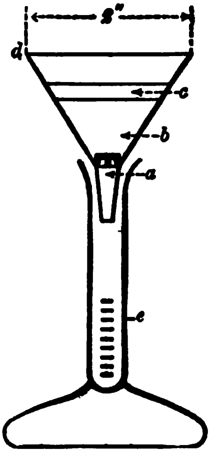

FIG. 82.

Application of the Test.—The thermometer is fixed so as to be inserted through the lid of the water bath, described before, into the water (which is to be steadily maintained at a temperature of 160° F.) to a depth of 2¾ inches. Fifty grains of nitro-glycerine, to be tested, are weighed out into a test tube in such a way as not to soil the sides of the tube. A test paper is fixed on the hook of the glass rod so that when inserted into the tube it will be in a vertical position. A sufficient amount of a mixture of half distilled water and half glycerine to moisten the upper half of the paper is now applied to the upper edge of the test paper by means of a camel's hair pencil, the cork carrying the rod and paper is fixed into the test tube and the position of the paper adjusted so that its lower edge is about half-way down the tube; the latter is then inserted through one of the perforations of the cover, to such a depth that the lower margin of the moistened part of the paper is about ⅝ of an inch above the surface of the cover. The test is complete when the faint brown line, which after a time makes its appearance at the line of boundary between the dry and moist part of the paper, equals in tint the brown line of the standard tint paper.

The nitro-glycerine under examination will not be considered as *thoroughly purified* within the terms of the licence unless the time necessary to produce the standard tint as above described IS AT LEAST 15 MINUTES.

B.—BLASTING GELATINE, GELATINE DYNAMITE, AND ANALOGOUS PREPARATIONS.

Fifty grains of blasting gelatine are to be intimately incorporated with 100 grains of French chalk. This may be readily effected by carefully working the two materials together with a wooden pestle in a wooden mortar. The mixture is to be gradually introduced into a test tube of the dimensions recommended above for the dynamite heat test, with the aid of gentle tapping upon the table, between the introduction of successive portions of the mixture into the tube, so that when the tube contains all the mixture it shall be filled to the extent of 1¾ inch of its height. The test paper is then to be inserted, and the heat is to be applied in the manner prescribed above for the dynamite heat test, and the sample tested as to withstand exposure to 160° F. for a period of ten minutes, before producing a discoloration of the test papers corresponding in tint to the standard colour test which is employed for governing the results of the dynamite heat test.

Testing Gun-Cotton, Schultze Gunpowder, E. C. Powder, and other Explosives of the 2nd Division of the Nitro-Compound Class.

A.—COMPRESSED GUN-COTTON, TONITE, &c., &c.

Sufficient material to serve for two or more tests is removed from the centre of the cartridge by gentle scraping, and, if necessary, further reduced by rubbing between the fingers.

The fine powder thus produced is spread out in a thin layer upon a paper tray 6 inches by 4½ inches, which is then placed inside a water oven, kept as nearly as possible at 120° F.

The wire gauze shelves in the oven should be about 3 inches apart. The sample is allowed to remain at rest for fifteen minutes in the oven, the door of which is left wide open.

After the lapse of fifteen minutes the tray is removed and exposed to the air of the room for two hours, the sample being at some point within that time rubbed upon the tray with the hand, in order to reduce it to a fine and uniform state of division.

Application of the Test.—The cover of the water bath is fitted with the gas regulator which is inserted through the centre hole (No. 4). The thermometer is fixed into hole No. 3. The water in the bath is then heated to 150° F., and the regulator set to maintain that temperature.

Twenty grains of the sample to be tested are weighed out, placed in the test tube, and gently pressed down until the specimen occupies a space of as nearly as possible $1\frac{6}{10}$ inch in a test tube of the dimensions specified.

A test paper is affixed to the hook of the glass rod or tube, and moistened by touching the upper edge with a drop of distilled water containing 50 per cent. of Price's glycerine. The quantity of liquid used must be only sufficient to moisten about half of the paper.

The cork carrying the rod and test paper is then fixed into the test tube, and the latter inserted into the bath to a depth of 2½ inches, measured from the cover, the regulator and thermometer being inserted to the same depth. The test paper is to be kept near the top of the test tube, but clear of the cork, until the tube has been immersed for about five minutes.

A ring of moisture will about this time be deposited upon the sides of the test tube a little above the cover of the bath; the glass rod must then be lowered until the lower margin of the moistened part of the paper is on a level with the bottom of the ring of moisture in the tube. The paper is now closely watched.

The test is complete when a very faint brown coloration makes its appearance at the line of boundary between the dry and moist parts of the paper.

The interval of time between the first insertion of the tube containing the sample of gun-cotton in the water at 150° and the first appearance of discoloration on the paper constitutes the test, and this interval of time must be *not less* than ten minutes, or the sample will not be accepted as properly purified.

B.—SCHULTZE POWDER, E. C. POWDER, COLLODION-COTTON, &c., &c.

Sufficient of the sample, without further mechanical division, is dried in the oven as above, and then exposed for two hours to the air. The test as directed above for compressed gun-cotton, &c., is then applied, the minimum duration of test being the same, viz., ten minutes.

Exudation and Liquefaction Test for Blasting Gelatine, Gelatine Dynamite, and Analogous Preparations.

Test for Liquefaction.—A cylinder of blasting gelatine is to be cut from the cartridge to be tested, the length of the cylinder to be about equal to its diameter, and the ends being cut flat.

The cylinder is to be placed on end on a flat surface without any wrapper, and secured by a pin passing vertically through its centre.

In this condition the cylinder is to be exposed for 144 consecutive hours (six days and nights) to a temperature ranging from 85° to 90° Fahrenheit (inclusive), and during such exposure the cylinder shall not diminish in height by more than one-fourth of its original height, and the upper cut surface shall retain its flatness and the sharpness of its edge.

Note.—If the blasting gelatine and the gelatine dynamite to be tested be not made up in a cylindrical form, the above test is to be applied with the necessary modifications.

Test for Liability of Exudation.—There shall be no separation from the general mass of the blasting gelatine or gelatine dynamite of a substance of less consistency than the bulk of the remaining portion of the material, under any conditions of storage, transport, or use, or when the material is subjected three times in succession to alternate freezing and thawing, or when subjected to the liquefaction test hereinbefore described.

APPENDIX.

Symbols and Atomic Weights of the Elements.

Element.	Symbol.	Atomic wt.	Observer.
Aluminium	Al	27·02	Mallet.
Antimony	Sb	120·00	Schneider, Cooke.
Arsenic	As	75·15	Kessler.
Barium	Ba	136·84	Marignac.
Bismuth	Bi	210·00	Dumas.
Boron	B	11·04	Berzelius.
Bromine	Br	79·76	Stas.
Cadmium	Cd	112·04	Leussen.
Cæsium	Cs	133·00	Bunsen.
Calcium	Ca	39·90	Erdmann.
Carbon	C	11·97	Dumas, Liebig.
Cerium	Ce	138·24	Rammelsberg.
Chlorine	Cl	35·37	Stas.
Chromium	Cr	52·08	Siewart.
Cobalt	Co	58·74	Russell.
Copper	Cu	63·12	Millon & Commaille.
Didymium	D	142·44	Hermann.
Erbium	E	168·90	Bahr & Bunsen.
Fluorine	F	18·96	Luca, Louyet.
Gallium	Ga	69·80	Lecoq de Boisbaudran.
Glucinum	Gl	9·30	Awdejew, Klatzo.
Gold	Au	196·71	Berzelius.
Hydrogen	H	1·00	Dulong & Berzelius.
Indium	In	113·40	Winkler, Bunsen.

Element.	Symbol.	Atomic wt.	Observer.
Iodine	I	126·54	Stas.
Iridium	Ir	196·87	Berzelius.
Iron	Fe	56·00	Dumas.
Lanthanum	La	139·33	Hermann.
Lead	Pb	206·40	Stas.
Lithium	Li	7·00	Stas.
Magnesium	Mg	23·94	Dumas.
Manganese	Mn	54·04	Schneider.
Mercury	Hg	200·00	Erdmann.
Molybdenum	Mo	96·00	Dumas, Debray.
Nickel	Ni	58·74	Russell.
Niobium	Nb	94·00	Marignac.
Nitrogen	N	14·01	Stas.
Osmium	Os	199·03	Berzelius.
Oxygen	O	15·96	
Palladium	Pd	106·57	Berzelius.
Phosphorus	P	30·96	Schrötter.
Platinum	Pt	194·38	Seubert.
Potassium	K	39·04	Stas.
Rhodium	Rh	104·21	Berzelius.
Rubidium	Rb	85·40	Bunsen, Piccard.
Ruthenium	Ru	104·40	Berzelius.
Selenium	Se	79·46	Dumas.
Silver	Ag	107·67	Stas.
Silicon	Si	28·10	Dumas.
Sodium	Na	22·99	Stas.
Strontium	Sr	87·54	Marignac.
Sulphur	S	31·996	Stas.
Tantalum	Ta	182·300	Marignac.
Tellurium	Te	128·06	V. Hauer.
Thallium	Tl	203·66	Crookes.
Thorium	Th	231·44	Delafontaine.
Tin	Sn	118·10	Dumas.
Titanium	Ti	50·00	Pierre.
Tungsten	W	184·00	Schneider, Roscoe.
Uranium	U	237·60	Ebelman.
Vanadium	V	51·35	Roscoe.
Yttrium	Y	92·55	Bahr & Bunsen.
Zinc	Zn	65·16	Oxel Erdmann.
Zirconium	Zr	89·60	Marignac.

Table of Atomic Weights.

(*Issued December* 6, 1890.)

Revised for the Committee of Revision and Publication of the Pharmacopœia of the United States of America. By F. W. Clarke, Chief Chemist of the United States Geological Survey.

This table represents the latest and most trustworthy results, reduced to a uniform basis of comparison, with oxygen = 16 as starting point of the system. No decimal places representing large uncertainties are used. When values vary, with equal probability on both sides, so far as our present knowledge goes, as in the case of a cadmium (111·8 and 112·2), the mean value is given in the Table.

The names of elements occurring in pharmacopœial, medicinal, chemicals, are printed in italics.

Name.	Symbol.	Atomic weight.
Aluminium	*Al*	27·
Antimony	*Sb*	120·
Arsenic	*As*	75·
Barium	*Ba*	137·
Bismuth	*Bi*	208·9
Boron	*B*	11·
Bromine	*Br*	79·95
Cadmium	Cd	112·
Cæsium	Cs	132·9
Calcium	*Ca*	40·
Carbon	*C*	12·
Cerium	*Ce*	140·2
Chlorine	*Cl*	35·45
Chromium	*Cr*	52·1
Cobalt	Co	59·
*Columbium	Cb	94·
Copper	*Cu*	63·4
†Didymium	Di	142·3
Erbium	Er	166·3
Fluorine	F	19·
Gallium	Ga	69·
Germanium	Ge	72·3
‡Glucinum	Gl	9·
Gold	*Au*	197·3
Hydrogen	*H*	1·007
Indium	In	113·7

* Has priority over niobium.

† Now split into neo- and phraseo-didymium.

‡ Has priority over beryllium.

Name.	Symbol.	Atomic weight.
Iodine	*I*	126·85
Iridium	Ir	193·1
Iron	*Fe*	56·
Lanthanum	La	138·2
Lead	*Pb*	206·95
Lithium	*Li*	7·02
Magnesium	*Mg*	24·3
Manganese	*Mn*	55·
Mercury	*Hg*	200·
Molybdenum	Mo	96·
Nickel	Ni	58·7
Nitrogen	*N*	14·03
Osmium	Os	191·7
§*Oxygen*	*O*	16·
Palladium	Pd	106·6
Phosphorus	*P*	31·
Platinum	Pt	195·
Potassium	*K*	39·11
Rhodium	Rh	103·5
Rubidium	Rb	85·5
Ruthenium	Ru	101·6
Samarium	Sm	150·
Scandium	Sc	44·
Selenium	Se	79·
Silicon	*Si*	28·4
Silver	*Ag*	107·92
Sodium	*Na*	23·05
Strontium	Sr	87·6
Sulphur	*S*	32·06
Tantalum	Ta	182·6
Tellurium	Te	125·
Terbium	Tb	159·5
Thallium	Tl	204·18
Thorium	Th	232·6
Tin	Sn	119·
Titanium	Ti	48
Tungsten	W	184·
Uranium	U	239·6
Vanadium	V	51·4
Ytterbium	Yb	173·
Yttrium	Yt	89·1
Zinc	*Zn*	65·3
Zirconium	Zr	90·6

§ Standard, or basis of the system.

Factors for Ascertaining the Amount of Constituent Sought from the Form in which it is Weighed.

Element.	Form.	Sought.	Factor.
Aluminium	Al_2O_3	Al_2	·5340
Ammonium	NH_4Cl	NH_3	·3180
,,	$2\,NH_4Cl,PtCl_4$	$2\,NH_3$	·0761
Antimony	Sb_2O_3	Sb_2	·8356
,,	Sb_2S_3	Sb_2	·7177
,,	Sb_2S_3	Sb_2O_3	·8588
,,	Sb_2O_4	Sb_2O_3	·9481
Arsenic	As_2O_3	As_2	·7576
,,	As_2O_5	As_2	·6522
,,	As_2O_5	As_2O_3	·8609
,,	As_2S_3	As_2O_3	·8049
,,	As_2S_3	As_2O_5	·9350
,,	$(MgAmAsO_4)_2OH_2$	As_2O_5	·6053
,,	,,	As_2O_3	·5211
Barium	BaO	Ba	·8954
,,	$BaSO_4$	BaO	·6567
,,	$BaCO_3$	BaO	·7767
,,	BaF_2,SiF_4	BaO	·5484
Bismuth	Bi_2O_3	Bi_2	·8966
Boron	B_2O_3	B_2	·3143
Bromine	$AgBr$	Br	·4256
Cadmium	CdO	Cd	·8750
Calcium	CaO	Ca	·7143
,,	$CaSO_4$	CaO	·4118
,,	$CaCO_3$	CaO	·5600
Carbon	CO_2	C	·2727
,,	$CaCO_3$	CO_2	·4400
Chlorine	$AgCl$	Cl	·2472
,,	$AgCl$	HCl	·2542
Chromium	Cr_2O_3	Cr_2	·6862
,,	Cr_2O_3	$2\,CrO_3$	1·3138
,,	$PbCrO_4$	CrO_3	·3106
Cobalt	Co	CoO	1·2712
,,	$Co_{12}O_{19}$	Co_{12}	·6999
,,	Co_3O_5	Co_3	·6887
,,	$CoSO_4$	CoO	·4839
,,	Co_3O_4	Co_3	·7344
,,	$\left\{\begin{matrix} Co_2O_3,\ 3\,K_2O, \\ 5\,N_2O_3,\ 2\,H_2O \end{matrix}\right\}$	$2\,CoO$	·1735
,,	$2\,CoSO_4 + 3\,K_2SO_4$	$2\,CoO$	·1802
,,	,,	Co_2	·1417

Element.	Form.	Sought.	Factor.
Copper	CuO	Cu	·7985
,,	Cu_2S	Cu_2	·7985
Fluorine	CaF_2	F_2	·4872
,,	SiF_4	F_4	·7308
Hydrogen	H_2O	H_2	·1111
Iodine	AgI	I	·5405
,,	PdI_2	I_2	·7056
Iron	Fe_2O_3	Fe_2	·7000
,,	Fe_2O_3	$2\ FeO$	·9000
,,	FeS	Fe	·6364
Lead	PbO	Pb	·9283
,,	$PbSO_4$	PbO	·7360
,,	$PbSO_4$	Pb	·6832
,,	$PbCl_2$	Pb	·7448
,,	$PbCl_2$	PbO	·8024
,,	PbS	PbO	·9331
Lithium	Li_2CO_3	Li_2O	·4054
,,	Li_2SO_4	Li_2O	·2727
,,	Li_3PO_4	Li_2O	·3879
Magnesium	MgO	Mg	·6000
,,	$MgSO_4$	MgO	·3335
,,	$Mg_2P_2O_7$	$2\ MgO$	·3604
Manganese	MnO	Mn	·7747
,,	Mn_3O_4	Mn_3	·7205
,,	Mn_2O_3	Mn_2	·6962
,,	$MnSO_4$	MnO	·4702
,,	MnS	MnO	·8161
,,	MnS	Mn	·6322
Mercury	$2\ Hg$	Hg_2O	1·0400
,,	Hg	HgO	1·0800
,,	Hg_2Cl_2	$2\ Hg$	·8494
,,	HgS	Hg	·8621
Nickel	NiO	Ni	·7867
Nitrogen	$2\ AmCl, PtCl_4$	N_2	·0627
,,	Pt	N_2	·1416
,,	$BaSO_4$	N_2O_5	·4635
,,	$AgCN$	CN	·1941
,,	$AgCN$	HCN	·2016
Oxygen	Al_2O_3	O_3	·4660
,,	Sb_2O_3	O_3	·1644
,,	As_2O_3	O_3	·2424
,,	As_2O_5	O_5	·3478
,,	BaO	O	·1046
,,	Bi_2O_3	O_3	·1035

Element.	Form.	Sought.	Factor.
Oxygen	CdO	O	·1250
,,	Cr_2O_3	O_3	·3138
,,	CoO	O	·2133
,,	CuO	O	·2015
,,	FeO	O	·2222
,,	Fe_2O_3	O_3	·3000
,,	PbO	O	·0718
,,	CaO	O	·2857
,,	MgO	O	·3997
,,	MnO	O	·2254
,,	Mn_3O_4	O_4	·2795
,,	Mn_2O_3	O_3	·3038
,,	Hg_2O	O	·0385
,,	HgO	O	·0741
,,	NiO	O	·2133
,,	K_2O	O	·1698
,,	SiO_2	O_2	·5333
,,	Ag_2O	O	·0690
,,	Na_2O	O	·2581
,,	SrO	O	·1546
,,	SnO_2	O	·2133
,,	H_2O	O	·8889
,,	ZnO	O	·1974
Phosphorus	P_2O_5	P_2	·4366
,,	$Mg_2P_2O_7$	P_2O_5	·6396
,,	MgP_2O_7	$2\ PO_4$	·8559
,,	$Fe_2P_2O_8$	P_2O_5	·4702
,,	P_2O_5	$2\ PO_4$	1·3380
,,	Ag_3PO_4	$(P_2O_5)\frac{1}{2}$	·1695
,,	$U_4P_2O_{11}$	P_2O_5	·1991
,,	$Ag_4P_2O_7$	P_2O_5	·2344
Potassium	K_2O	K_2	·8302
,,	K_2SO_4	K_2O	·5400
,,	KNO_3	$(K_2O)\frac{1}{2}$	·4659
Potassium	KCl	K	·5245
,,	KCl	$(K_2O)\frac{1}{2}$	·6317
,,	$2\ KCl, PtCl_4$	K_2O	·1927
,,	,,	$2\ KCl$	·3070
Silicon	SiO_2	Si	·4667
Silver	$AgCl$	Ag	·7528
,,	$AgCl$	$(Ag_2O)\frac{1}{2}$	·8085
Sodium	Na_2O	Na_2	·7410
,,	Na_2SO_4	Na_2O	·4360
,,	$NaNO_3$	$(Na_2O)\frac{1}{2}$	·3647

Element.	Form.	Sought.	Factor.
Sodium	$NaCl$	$(Na_2O)\frac{1}{2}$	·5302
„	$NaCl$	Na	·3934
„	Na_2CO_3	Na_2O	·5849
Strontium	SrO	Sr	·8454
„	$SrSO_4$	SrO	·5640
„	$SrCO_3$	SrO	·7017
Sulphur	$BaSO_4$	S	·1373
„	As_2S_3	S_3	·3902
„	$BaSO_4$	SO_3	·3434
„	SO_3	SO_4	1·2000
Tin	SnO_2	Sn	·7867
„	SnO_2	SnO	·8933
Zinc	ZnO	Zn	·8026
„	ZnS	ZnO	·8352
„	ZnS	Zn	·6703

English Weights and Measures.

AVOIRDUPOIS.

	Grains.	Drachms.	Ounces.	Lbs.	Qrs.	Cwts.	Tons.
Grain .	1						
Drachm	27·34	1					
Ounce .	437·5	16	1				
Pound .	7,000·	256	16	1			
Quarter	196,000·	7,168	448	28	1		
Cwt. .	784,000·	28,672	1,792	112	4	1	
Ton .	15,680,000·	573,440	35,840	2,240	80	20	1

TROY WEIGHT.

	Grains.	Dwts.	Ounces.	Lb.
Grain . .	1			
Pennyweight .	24	1		
Ounce . .	480	20	1	
Pound . .	5,760	240	12	1

Cubic inches.
1 gallon = 277·276
1 pint = 34·659
1 fluid ounce = 1·7329
1 litre = 61·02705
1 cubic centimetre = 0·06102705
1 cubic inch = 16·386176 cubic centimetres.

1 cubic inch of distilled water in air at 62° F = 252·336 grains.
1 cubic inch ,, ,, *in vacuo* at 62° F = 252·645 grains.

1 minim is the volume of 0·91 grain of water.
1 fluid drachm ,, of 54·68 grains of water.
1 fluid ounce ,, of 437·5 grains of water.
1 gallon is the ,, of 10 pounds or 70,000 grains of water.

Weights and Measures of the British Pharmacopœia of 1867.

WEIGHTS.

1 grain, gr.
1 ounce, oz. = 437·5 grains.
1 pound = 16 ozs. = 7,000· ,,

MEASURES OF CAPACITY.

1 minim, min.
1 fluid drachm, fl. drm. = 60 minims.
1 fluid ounce, fl. oz. = 8 fluid drachms.
1 pint, O = 20 fluid ounces.
1 gallon, C = 8 pints.

MEASURES OF LENGTH.

1 line = $\frac{1}{12}$ inch.
1 inch = $\frac{1}{39·1393}$ seconds—pendulum.
12 ,, = 1 foot.
36 ,, = 3 feet = 1 yard.

Weights and Measures of the Metrical System.

WEIGHTS.

1 milligram = ·001 grm.
1 centigram = ·01 ,,
1 decigram = 0·1 ,,
1 grm. = weight of a cubic centimetre of water at 4° C.
1 decagram = 10 grms.
1 hectogram = 100 ,,
1 kilogram = 1,000 ,,

MEASURES OF CAPACITY.

1 millilitre = 1 cubic centimetre of water at 4° C.
1 centilitre = 10 cubic centimetres.
1 decilitre = 100 ,, ,,
1 litre = 1,000 ,, ,,

MEASURES OF LENGTH.

1 millimetre = ·001 metre.
1 centimetre = ·01 ,,
1 decimetre = 0·1 ,,
1 metre = the ten millionth part of a quarter of the earth's meridian (nearly).

Tables for Conversion of Metrical and English Measures

A. LENGTH.

METRICAL TO ENGLISH.		ENGLISH TO METRICAL.	
(1) Millimetres to inches.	(2) Metres to feet.	(3) Inches to millimetres.	(4) Feet to metres.
1 = 0·03937079	1 = 3·2808992	1 = 25·39954	1 = 0·30479449
2 = 0·07874158	2 = 6·5617984	2 = 50·79908	2 = 0·60958898
3 = 0·11811237	3 = 9·8426976	3 = 76·19862	3 = 0·91438347
4 = 0·15748316	4 = 13·1235968	4 = 101·59816	4 = 1·21917796
5 = 0·19685395	5 = 16·4044960	5 = 126·99770	5 = 1·52397245
6 = 0·23622474	6 = 19·6853952	6 = 152·39724	6 = 1·82876694
7 = 0·27559553	7 = 22·9662944	7 = 177·79678	7 = 2·13356143
8 = 0·31496632	8 = 26·2471936	8 = 203·19632	8 = 2·43835592
9 = 0·35433711	9 = 29·5280928	9 = 228·59586	9 = 2·74315041

B. CAPACITY.

METRICAL TO ENGLISH.

(1) Cubic centimetres to cubic inches.	(2) Litres to fluid ounces.	(3) Litres to pints.	(4) Litres to gallons.
1 = 0·06102705	1 = 35·215468	1 = 1·7607734	1 = 0·22009668
2 = 0·12205410	2 = 70·430936	2 = 3·5215468	2 = 0·44019336
3 = 0·18308115	3 = 105·646404	3 = 5·2823202	3 = 0·66029004
4 = 0·24410820	4 = 140·861872	4 = 7·0430936	4 = 0·88038672
5 = 0·30513525	5 = 176·077340	5 = 8·8038670	5 = 1·10048340
6 = 0·36616230	6 = 211·292808	6 = 10·5646404	6 = 1·32058008
7 = 0·42718935	7 = 246·508276	7 = 12·3254138	7 = 1·54067676
8 = 0·48821640	8 = 281·723744	8 = 14·0861872	8 = 1·76077344
9 = 0·54924345	9 = 316·939212	9 = 15·8469606	9 = 1·98087012

ENGLISH TO METRICAL.

(1) Cubic inches to cubic centimetres.	(2) Fluid ounces to cubic centimetres.	(3) Pints to litres.	(4) Gallons to litres.
1 = 16·386176	1 = 28·396612	1 = 0·567932	1 = 4·543458
2 = 32·772352	2 = 56·793224	2 = 1·135864	2 = 9·086916
3 = 49·158528	3 = 85·189856	3 = 1·703796	3 = 13·630374
4 = 65·544704	4 = 113·586448	4 = 2·271728	4 = 18·173832
5 = 81·930880	5 = 141·983060	5 = 2·839660	5 = 22·717290
6 = 98·317056	6 = 170·379672	6 = 3·407592	6 = 27·270748
7 = 114·703232	7 = 198·776284	7 = 3·975524	7 = 31·804206
8 = 131·089408	8 = 227·172896	8 = 4·543456	8 = 36·347664
9 = 147·475584	9 = 255·569508	9 = 5·111388	9 = 40·891122

C. WEIGHT.

METRICAL TO ENGLISH.

(1) Grammes to grains.	(2) Kilogrammes to ounces.	(3) Kilogrammes to pounds.
1 = 15·4323488	1 = 35·27394	1 = 2·20462
2 = 30·8646976	2 = 70·54788	2 = 4·40924
3 = 46·2970464	3 = 105·82182	3 = 6·61386
4 = 61·7293952	4 = 141·09576	4 = 8·81848
5 = 77·1617440	5 = 176·36970	5 = 11·02310
6 = 92·5940928	6 = 211·64364	6 = 13·22772
7 = 108·0264416	7 = 246·91758	7 = 15·43234
8 = 123·4587904	8 = 282·19152	8 = 17·63696
9 = 138·8911392	9 = 317·46546	9 = 19·84158

ENGLISH TO METRICAL.

(1) Grains to grammes.	(2) Ounces to grammes.	(3) Pounds to kilogrammes.	(4) Hundredweights to kilogrammes.
1 = 0·0647989	1 = 28·34954	1 = 0·45359265	1 = 50·8023768
2 = 0·1295978	2 = 56·69908	2 = 0·90718530	2 = 101·6047536
3 = 0·1943967	3 = 85·04862	3 = 1·36077795	3 = 152·4071304
4 = 0·2591956	4 = 113·39816	4 = 1·81437060	4 = 203·2095072
5 = 0·3239945	5 = 141·74770	5 = 2·26796325	5 = 254·0118840
6 = 0·3887934	6 = 170·09724	6 = 2·72155590	6 = 304·8142608
7 = 0·4535923	7 = 198·44678	7 = 3·17514855	7 = 355·6166376
8 = 0·5183912	8 = 226·79632	8 = 3·62874120	8 = 406·4190144
9 = 0·5831901	9 = 255·14586	9 = 4·08233385	9 = 457·2213912

Hydrometer Tables.

DENSITIES CORRESPONDING TO DEGREES OF BAUMÉ'S HYDROMETER FOR LIQUIDS HEAVIER THAN WATER.

° B.	Density.	° B.	Density.	° B.	Density.	° B.	Density.
0	1·000	19	1·152	38	1·357	57	1·652
1	1·007	20	1·161	39	1·370	58	1·671
2	1·014	21	1·171	40	1·383	59	1·691
3	1·022	22	1·180	41	1·397	60	1·711
4	1·029	23	1·190	42	1·410	61	1·732
5	1·036	24	1·199	43	1·424	62	1·753
6	1·044	25	1·210	44	1·438	63	1·774
7	1·052	26	1·221	45	1·453	64	1·796
8	1·060	27	1·231	46	1·468	65	1·819
9	1·067	28	1·242	47	1·483	66	1·846
10	1·075	29	1·253	48	1·498	67	1·872
11	1·083	30	1·264	49	1·514	68	1·897
12	1·091	31	1·275	50	1·530	69	1·921
13	1·100	32	1·286	51	1·546	70	1·946
14	1·108	33	1·297	52	1·563	71	1·974
15	1·116	34	1·309	53	1·580	72	2·000
16	1·125	35	1·320	54	1·597	73	2·031
17	1·134	36	1·332	55	1·615	74	2·059
18	1·143	37	1·345	56	1·634		

Densities corresponding to Baumé's Hydrometer for Liquids Lighter than Water. (Francœur).

° B.	Density.	° B.	Density.	° B.	Density.	° B.	Density.
10	1·000	23	0·918	36	0·849	49	0·789
11	0·993	24	0·913	37	0·844	50	0·785
12	0·986	25	0·907	38	0·839	51	0·781
13	0·980	26	0·901	39	0·834	52	0·777
14	0·973	27	0·896	40	0·830	53	0·773
15	0·967	28	0·890	41	0·825	54	0·768
16	0·960	29	0·885	42	0·820	55	0·764
17	0·954	30	0·880	43	0·816	56	0·760
18	0·948	31	0·874	44	0·811	57	0·757
19	0·942	32	0·869	45	0·807	58	0·753
20	0·936	33	0·864	46	0·802	59	0·749
21	0·930	34	0·859	47	0·798	60	0·745
22	0·924	35	0·854	48	0·794		

Degrees on Twaddle's Hydrometer with the corresponding Densities.

° Tw.	Density.	° Tw.	Density.	° Tw.	Density.	° Tw.	Density.
1	1·005	8	1·040	15	1·075	22	1·110
2	1·010	9	1·045	16	1·080	23	1·115
3	1·015	10	1·050	17	1·085	24	1·120
4	1·020	11	1·055	18	1·090	25	1·125
5	1·025	12	1·060	19	1·095	26	1·130
6	1·030	13	1·065	20	1·100	27	1·135
7	1·035	14	1·070	21	1·105	28	1·140

Degrees Twaddle are converted into the corresponding specific gravities by multiplying them by 5 and adding 1,000.

Comparison of Thermometers.

Conversion of Thermometer Degrees.

° C. to ° R. : multiply by 4 and divide by 5.
° C. to ° F. : multiply by 9, divide by 5, then add 32.
° R. to ° C. : multiply by 5 and divide by 4.
° R. to ° F. : multiply by 9, divide by 4, then add 32.
° F. to ° R. : first subtract 32, then multiply by 4, and divide by 9.
° F. to ° C. : first subtract 32, then multiply by 5 and divide by9.

Comparison of Centigrade and Fahrenheit Degrees.

° C.	° F.	° C.	° F.	° C.	° F.	° C.	° F.
+500	+932	+74	+165·2	+45	+113	+16	+60·8
400	752	73	163·4	44	111·2	15	59
300	572	72	161·6	43	109·4	14	57·2
200	392	71	159·8	42	107·6	13	55·4
100	212	70	158	41	105·8	12	53·6
99	210·2	69	156·2	40	104	11	51·8
98	208·4	68	154·4	39	102·2	10	50
97	206·6	67	152·6	38	100·4	9	48·2
96	204·8	66	150·8	37	98·6	8	46·4
95	203	65	149	36	96·8	7	44·6
94	201·2	64	147·2	35	95	6	42·8
93	199·4	63	145·4	34	93·2	5	41
92	197·6	62	143·6	33	91·4	4	39·2
91	195·8	61	141·8	32	89·6	3	37·4
90	194	60	140	31	87·8	2	35·6
89	192·2	59	138·2	30	86	1	33·8
88	190·4	58	136·4	29	84·2	0	32
87	188·6	57	134·6	28	82·4	—1	30·2
86	186·8	56	132·8	27	80·6	2	28·4
85	185	55	131	26	78·8	3	26·6
84	183·2	54	129·2	25	77	4	24·8
83	181·4	53	127·4	24	75·2	5	23
82	179·6	52	125·6	23	73·4	6	21·2
81	177·8	51	123·8	22	71·6	7	19·4
80	176	50	122	21	69·8	8	17·6
79	174·2	49	120·2	20	68	9	15·8
78	172·4	48	118·4	19	66·2	10	14
77	170·6	47	116·6	18	64·4	11	12·2
76	168·8	46	114·8	17	62·6	12	10·4
75	167						

Percentages and Gravity of Sulphuric Acid.

TABLE SHOWING PERCENTAGES OF REAL SULPHURIC ACID (H_2SO_4) CORRESPONDING TO VARIOUS SPECIFIC GRAVITIES OF AQUEOUS SULPHURIC ACID.

Bineau; Otto. Temp. 15° C.

Specific Gravity.	Per cent.	Specific Gravity.	Per cent.	Specific Gravity.	Per cent.	Specific Gravity.	Per cent.
1·8426	100	1·675	75	1·398	50	1·182	25
1·842	99	1·663	74	1·3886	49	1·174	24
1·8400	98	1·651	73	1·379	48	1·167	23
1·840	97	1·639	72	1·370	47	1·159	22
1·8384	96	1·627	71	1·361	46	1·1516	21
1·8376	95	1·615	70	1·351	45	1·144	20
1·8356	94	1·604	69	1·342	44	1·136	19
1·834	93	1·592	68	1·333	43	1·129	18
1·831	92	1·580	67	1·324	42	1·121	17
1·827	91	1·568	66	1·315	41	1·1136	16
1·822	90	1·557	65	1·306	40	1·106	15
1·816	89	1·545	64	1·2976	39	1·098	14
1·809	88	1·534	63	1·289	38	1·091	13
1·802	87	1·523	62	1·281	37	1·083	12
1·794	86	1·512	61	1·272	36	1·0756	11
1·786	85	1·501	60	1·264	35	1·068	10
1·777	84	1·490	59	1·256	34	1·061	9
1·767	83	1·480	58	1·2476	33	1·0536	8
1·756	82	1·469	57	1·239	32	1·0464	7
1·745	81	1·4586	56	1·231	31	1·039	6
1·734	80	1·448	55	1·223	30	1·032	5
1·722	79	1·438	54	1·215	29	1·0256	4
1·710	78	1·428	53	1·2066	28	1·019	3
1·698	77	1·418	52	1·198	27	1·013	2
1·686	76	1·408	51	1·190	26	1·0064	1

Percentages and Gravity of Hydrochloric Acid.

TABLE GIVING THE PERCENTAGES OF HYDROCHLORIC ACID CONTAINED IN AQUEOUS SOLUTIONS OF THE GAS OF VARIOUS SPECIFIC GRAVITIES.

Ure. Temp. 15° C.

Specific Gravity.	HCl Per cent.	Specific Gravity.	HCl Per cent.	Specific Gravity.	HCl Per cent.	Specific Gravity.	HCl Per cent.
1·200	40·777	1·1515	30·582	1·1000	20·388	1·0497	10·194
1·1982	40·369	1·1494	30·174	1·0980	19·980	1·0477	9·786
1·1964	39·961	1·1473	29·767	1·0960	19·572	1·0457	9·379
1·1946	39·554	1·1452	29·359	1·0939	19·165	1·0437	8·971
1·1928	39·146	1·1431	28·951	1·0919	18·757	1·0417	8·563
1·1910	38·738	1·141	28·544	1·0899	18·349	1·0397	8·155
1·1893	38·330	1·1389	28·136	1·0879	17·941	1·0377	7·747
1·1875	37·923	1·1369	27·728	1·0859	17·534	1·0357	7·340
1·1857	37·516	1·1349	27·321	1·0838	17·126	1·0337	6·932
1·1846	37·108	1·1328	26·913	1·0818	16·718	1·0318	6·524
1·1822	36·700	1·1308	26·505	1·0798	16·310	1·0298	6·116
1·1802	36·292	1·1287	26·098	1·0778	15·902	1·0279	5·709
1·1782	35·884	1·1267	25·690	1·0758	15·494	1·0259	5·301
1·1762	35·476	1·1247	25·282	1·0738	15·087	1·0239	4·893
1·1741	35·068	1·1226	24·874	1·0718	14·679	1·0220	4·486
1·1721	34·660	1·1206	24·466	1·0697	14·271	1·0200	4·078
1·1701	34·252	1·1185	24·058	1·0677	13·863	1·0180	3·670
1·1681	33·845	1·1164	23·650	1·0657	13·456	1·0160	3·262
1·1661	33·437	1·1143	23·242	1·0637	13·049	1·0140	2·854
1·1641	33·029	1·1123	22·834	1·0617	12·641	1·0120	2·447
1·1620	32·621	1·1102	22·426	1·0597	12·233	1·0100	2·039
1·1599	32·213	1·1082	22·019	1·0577	11·825	1·0080	1·631
1·1578	31·805	1·1061	21·611	1·0557	11·418	1·0060	1·124
1·1557	31·398	1·1041	21·203	1·0537	11·010	1·0040	·816
1·1536	30·990	1·1020	20·796	1·0517	10·602	1·0020	·408

Percentages and Gravity of Nitric Acid.

TABLE SHOWING THE PERCENTAGES OF NITRIC ACID (HNO_3) IN AQUEOUS SOLUTIONS OF VARIOUS SPECIFIC GRAVITIES.

Kolb, Ann. Ch. Phys. (4) 136. Temp. 15° C.

HNO_3 Per cent.	Specific Gravity.	HNO_3 Per cent.	Specific Gravity.	HNO_3 Per cent.	Specific Gravity.	HNO_3 Per cent.	Specific Gravity.
100·00	1·530	80·96	1·463	59·59	1·372	39·00	1·244
99·84	1·530	80·00	1·460	58·88	1·368	37·95	1·237
99·72	1·530	79·00	1·456	58·00	1·363	36·00	1·225
99·52	1·529	77·66	1·451	57·00	1·358	35·00	1·218
97·89	1·523	76·00	1·445	56·10	1·353	33·86	1·211
97·00	1·520	75·00	1·442	55·00	1·346	32·00	1·198
96·00	1·516	74·01	1·438	54·00	1·341	31·00	1·192
95·27	1·514	73·00	1·435	53·81	1·339	30·00	1·185
94·00	1·509	72·39	1·432	53·00	1·335	29·00	1·179
93·01	1·506	71·24	1·429	52·33	1·331	28·00	1·172
92·00	1·503	69·96	1·423	50·99	1·323	27·00	1·166
91·00	1·499	69·20	1·419	49·97	1·317	25·71	1·157
90·00	1·495	68·00	1·414	49·00	1·312	23·00	1·138
89·56	1·494	67·00	1·410	48·00	1·304	20·00	1·120
88·00	1·488	66·00	1·405	47·18	1·298	17·47	1·105
87·45	1·486	65·07	1·400	46·64	1·295	15·00	1·089
86·17	1·482	64·00	1·395	45·00	1·284	13·00	1·077
85·00	1·478	63·59	1·393	43·53	1·274	11·41	1·067
84·00	1·474	62·00	1·386	42·00	1·264	7·22	1·045
83·00	1·470	61·21	1·381	41·00	1·257	4·00	1·022
82·00	1·467	60·00	1·374	40·00	1·251	2·00	1·010

Oxide of Potassium and Caustic Potash.

TABLE SHOWING THE PERCENTAGE OF OXIDE OF POTASSIUM (K_2O) IN AQUEOUS SOLUTIONS OF CAUSTIC POTASH (KHO) OF VARIOUS SPECIFIC GRAVITIES.

Tünnermann. Temp. 15° C.

Specific Gravity.	Per cent.	Specific Gravity.	Per cent.
1·3300	28·290	1·1437	14·145
1·3131	27·158	1·1308	13·013
1·2966	26 027	1·1182	11·882
1·2805	24·895	1·1059	10·750
1·2648	23·764	1·0938	9·619
1·2493	22 632	1·0819	8·487
1·2342	21·500	1·0703	7·355
1·2268	20·935	1·0589	6·224
1·2122	19·803	1·0478	5·002
1·1979	18·671	1·0369	3·961
1·1839	17·540	1·0260	2·829
1·1702	16·408	1·0153	1·697
1·1568	15·277	1·0050	·5658

Oxide of Sodium and Caustic Soda.

TABLE SHOWING THE PERCENTAGE OF OXIDE OF SODIUM (Na_2O) IN AQUEOUS SOLUTIONS OF CAUSTIC SODA (NaHO) OF VARIOUS SPECIFIC GRAVITIES.

Tünnermann.

Specific Gravity.	Per cent.	Specific Gravity.	Per cent.	Specific Gravity.	Per cent.	Specific Gravity.	Per cent.
1·4285	30·220	1·3198	22·363	1·2392	15·110	1·1042	7·253
1·4193	29·616	1·3143	21·894	1·2280	14·500	1 0948	6·648
1·4101	29·011	1·3125	21·758	1·2178	13·901	1 0855	6·044
1·4011	28·407	1·3053	21·154	1·2058	13·297	1·0764	5·440
1·3923	27·802	1·2982	20·550	1·1948	12·692	1·0675	4·835
1·3836	27·200	1·2912	19·945	1·1841	12·088	1·0587	4·231
1·3751	26·594	1·2843	19·341	1·1734	11·484	1·0500	3·626
1·3668	25·989	1·2775	18·730	1·1630	10·879	1·0414	3·022
1·3586	25·385	1·2708	18·132	1·1528	10·275	1·0330	2·418
1·3505	24·780	1·2642	17·528	1·1428	9·670	1·0246	1·813
1·3426	24·176	1·2578	16·923	1·1330	9·066	1·0163	1·209
1·3349	23·572	1·2515	16·379	1·1233	8·462	1·0081	0·604
1·3273	22·967	1·2453	15·714	1·1137	7 857	1·0040	0·302

Percentages and Gravity of Ammonia.

Table showing the Percentages of Ammonia (NH_3) in Aqueous Solutions of the Gas of various Specific Gravities.

Carius. Temp. 15 ° C.

Specific gravity.	NH_3 Per cent.	Specific gravity.	NH_3 Per cent.	Specific gravity.	NH_3 Per cent.
0·8844	36	0·9133	24	0·9520	12
0·8864	35	0·9162	23	0·9556	11
0·8885	34	0·9191	22	0·9593	10
0·8907	33	0·9221	21	0·9631	9
0·8929	32	0·9251	20	0·9670	8
0·8953	31	0·9283	19	0·9709	7
0·8976	30	0·9314	18	0·9749	6
0·9001	29	0·9347	17	0·9790	5
0·9026	28	0·9380	16	0·9031	4
0·9052	27	0·9414	15	0·9873	3
0·9078	26	0·9449	14	0·9915	2
0·9106	25	0·9484	13	0·9959	1

Melting Points of the Metals.

	Melting point ° C.		Melting point ° C.
Aluminium	700	Lithium	180
Antimony	425	Magnesium	232
Arsenic	415	Mercury	−40
Bismuth	270	Nickel	1,500—1,600
Cadmium	320	Phosphorus	44
Cobalt	1,050—1,200	Potassium	62·5
Copper	1,050	Platinum	2,600
Gold	1,250	Silver	1,000
Indium	176	Selenium	217
Iron, Cast	1,050—1,200	Sodium	96
„ Steel	1,300—1,400	Sulphur	115
„ Wrought	1,500—1,600	Thallium	290
Lead	330	Tin	235
		Zinc	412

Comparative Wear of Axle-bearings.

(Thurston).

Bearing.	Composition.			Cost per 100 lbs.*	Miles run per lb.	Wear per 100 miles for four bearings.
	Copper.	Tin.	Antimony.			
				$ c.		grs.
Gun metal . . .	83	17	—	26 60	25,489	200
,, ,, . . .	82	18	—	28 68	27,918	252
White metal . .	3	90	7	32 85	22,075	366
,, ,, . . .	5	85	10	32 27	24,857	284
Lead composition: lead 84; antimony 16	..	..	..	13 04	22,921	308
Gun metal on brake cars	82	18	..	28 68	2,576	274

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* Including melting expenses, loss, &c. These figures are constantly varying.

Aqueous Vapour.

TENSION OF AQUEOUS VAPOUR IN MM. OF MERCURY.

t° C.	mm.	t° C.	mm.	t° C.	mm.	t° C.	Atmos.
—10	2·08	16	13·54	90	525·39	100	1·0
—9	2·26	17	14·42	95	633·69	110	1·4
—8	2·46	18	15·36	99	733·21	120	1·96
—7	2·67	19	16·35	99·1	735·85	130	2·67
—6	2·89	20	17·39	99·2	738·50	140	3·57
—5	3·13	21	18·50	99·3	741·16	150	4·7
—4	3·39	22	19·66	99·4	743·83	160	6·1
—3	3·66	23	20·89	99·5	746·50	170	7·8
—2	3·96	24	22·18	99·6	749·18	180	9·9
—1	4·27	25	23·55	99·7	751·87	190	12·4
0	4·60	26	24·99	99·8	754·57	200	15·4
1	4·94	27	26·51	99·9	757·28	210	18·8
2	5·30	28	28·10	100	760·00	220	22·9
3	5·69	29	29·78	100·1	762·73	230	27·5
4	6·10	30	31·55	100·2	765·46		
5	6·53	35	41·83	100·3	768·20		
6	7·00	40	54·91	100·4	771·95		
7	7·49	45	71·39	100·5	773·71		
8	8·02	50	91·98	100·6	776·48		
9	8·57	55	117·48	100·7	779·26		
10	9·17	60	148·79	100·8	782·04		
11	9·79	65	186·94	100·9	784·83		
12	10·46	70	233·08	101	787·63		
13	11·16	75	288·50	105	906·41		
14	11·91	80	354·62	110	1075·37		
15	12·69	85	433·00				

Text of the Petroleum Act, 1879.

(42 & 43 VICT. c. 47.)

1. This Act may be cited as the Petroleum Act, 1879.

This Act shall be construed as one with the Petroleum Act, 1871, and together with that Act may be cited as the Petroleum Acts, 1871 and 1879.

2. Whereas by the Petroleum Act, 1871, it is enacted that the term "petroleum to which this Act applies" means such of the petroleum defined by that Act as, when tested in manner set forth in Schedule One to that Act, gives off an inflammable vapour at a temperature of less than one hundred degrees of Fahrenheit's thermometer, and it is expedient to alter the said test: Be it therefore enacted that—

In the Petroleum Act, 1871, the term "petroleum to which this Act applies" shall mean such of the petroleum defined by section three of that Act as, when tested in manner set forth in Schedule One to this Act, gives off an inflammable vapour at a temperature of less than seventy-three degrees of Fahrenheit's thermometer.

Every reference in the Petroleum Act, 1871, to Schedule One to that Act shall be construed to refer to Schedule One to this Act.

3. A model of the apparatus for testing petroleum, as described in Schedule One to this Act, shall be deposited with the Board of Trade, and the Board of Trade shall, on payment of such fee, not exceeding five shillings, as they from time to time prescribe, cause to be compared with such model and verified every apparatus constructed in accordance with Schedule One to this Act which is submitted to them for the purpose, and if the same is found correct shall stamp the same with a mark approved of by the Board and notified in the London Gazette.

An apparatus for testing petroleum purporting to be stamped with the said mark shall, until the contrary is proved, be deemed to have been verified by the Board of Trade.

All fees under this section shall be paid into the Exchequer.

4. The Petroleum Act, 1871, shall continue in force until otherwise directed by Parliament.

5. This Act shall come into operation on the thirty-first day of December one thousand eight hundred and seventy-nine, which day is in this Act referred to as the commencement of this Act.

6. The Petroleum Act, 1871, shall be repealed after the

commencement of this Act to the extent in the third column of the Second Schedule to this Act mentioned.

Provided that any sample of petroleum taken before the commencement of this Act shall be tested in manner set forth in Schedule One to the Petroleum Act, 1871, and any offence committed before the commencement of this Act shall be prosecuted, and any investigation, legal proceeding, or remedy in relation to such offence, or to any act done before the commencement of this Act, shall be instituted, carried on, and have effect as if the provisions of this Act, other than those continuing the Petroleum Act, 1871, had not been passed.

FIRST SCHEDULE.—MODE OF TESTING PETROLEUM SO AS TO ASCERTAIN THE TEMPERATURE AT WHICH IT WILL GIVE OFF INFLAMMABLE VAPOUR. SPECIFICATION OF THE TEST APPARATUS. (See *ante*, p. 204.) DIRECTIONS FOR APPLYING THE FLASHING TEST. (See *ante*, p. 207.)

SECOND SCHEDULE.—ACT REPEALED.

Year and Chapter.	Title.	Extent of repeal.
34 & 35 Vict. c. 105.	The Petroleum Act, 1871.	Section three, from "and the term petroleum to which this Act applies" inclusive to the end of the section. Section eighteen.

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